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Acquisition and Chemical Analysis of Mother's Milk for Selected Toxic Substances

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16. ABSTRACT

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Fewer semivolatile compounds of interest were found. Polychlorinated naphthalenes, polybrominated biphenyls, chlorinated phenols, and other compounds were specifically sought and not detected (limit of detection about 20-100 ng/mL milk). Polychlorinated biphenyls (PCBs) and DDE were found.

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by

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ABSTRACT

Samples of mother's milk were collected from Bayonne, NJ; Jersey City, NJ; Pittsburgh, PA; Baton Rouge, LA; and Charleston, WV, and analyzed for volatile (purgeables) and semivolatile (extractable) organics using glass capillary gas chromatography/mass spectrometry/computer. In the volatile fraction, 26 halogenated hydrocarbons, 17 aldehydes, 20 ketones, 11 alcohols, 2 acids, 3 ethers, 1 epoxide, 14 furans, 26 other oxygenated compounds, 4 sulfur-containing compounds, 7 nitrogen-containing compounds, 13 alkanes, 12 alkenes, 7 alkynes, 11 cyclic hydrocarbons, and 15 aromatics were found, including major peaks for hexanal, limonene, dichlorobenzene, and some esters. The levels of dichlorobenzene appeared to be significantly higher in the samples from Jersey City and Bayonne than in samples from other sites. Jersey City samples also appeared to have significantly higher levels of tetrachloroethylene. Charleston and Jersey City samples appeared to have significantly higher levels of chloroform; however, chloroform was observed in the blanks at about 20% of that in the samples. Due to the small sample size and lack of control over the solicitation of sample donors, the data cannot be used to extrapolate to the general population.

Fewer semivolatile compounds of interest were found. Polychlorinated naphthalenes, polybrominated biphenyls, chlorinated phenols, and other compounds were specifically sought and not detected (limit of detection about 20-100 ng/mL milk). Polychlorinated biphenyls (PCBs) and DDE were found.

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LIST OF ABBREVIATIONS AND SYMBOLS

ABBREVIATIONS

DDT -- 1,1-Bis(p-chlorophenyl)-2,2-trichloroethane
dpm -- Disintegrations per minute
ECD -- Electon capture detection
GC -- Gas chromatography
MS -- Mass spectrometry (electron impact ionization)
NICIMS -- Negative ion chemical ionization mass spectrometry
OMB -- Office of Management and Budget
PBBs -- Polybrominated biphenyls
PCBs -- Polychlorinated biphenyls
PCF -- Participant Consent Form
PCN -- Polychlorinated Naphthalene
PLF -- Participant Listing Form
SQ -- Study Questionnaire

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SECTION 1
INTRODUCTION

BACKGROUND

It is becoming increasingly important to correlate ambient environmental pollutant levels with human body burden. Establishment of this correlation ("exposure assessment") may provide a link between pollution and health effects. This correlation is of interest for both scientific research and regulatory risk assessment.

Measurement of pollutant body burden levels generally requires invasive techniques (exceptions are breath and urine sampling) which are undesirable from the subjects' viewpoint. Some invasive techniques are generally regarded as acceptable (*e.g.*, blood samples), while others are generally considered unacceptable from living donors (*e.g.* adipose tissue, internal organs, etc.). Mother's milk is an attractive medium for several reasons: (1) sample collection is reasonably straightforward; (2) milk contains a high amount of fat (about 3.5 percent, as shown in see Table 1), so fat-soluble pollutants such as DDT and polychlorinated biphenyls (PCBs) are likely to be found in higher concentrations in milk than in blood or urine; (3) large (50-100 mL) volumes are easily collected for analysis, increasing analytical reliability and detection limit; and (4) the population of nursing mothers is large relative to pathology samples such as adipose tissue. In addition, an assessment of pollutant concentrations in mother's milk may be used to predict the pollutant intake by the nursing infant.

The major disadvantages of mother's milk as a human-sampling medium relate to the sampling demography: only young-to-middle-aged females are nursing. Thus, any use of mother's milk in a probability-based sampling framework extrapolated to the general population would be fraught with difficulties, such as locating donors.

Table 1. COMPARISON BETWEEN HUMAN AND COW'S MILK(1)

Parameter	Human Milk	Cow's Milk
Water and solid content	Same in both; 87 to 87.5 percent is water	
Calories	Same in both; 20 calories per ounce	
Protein	1 to 1.5 percent; 60 percent of this is lactalbumin and 40 percent casein	3.5 percent; 15 percent of this is lactalbumin and 85 percent casein
Carbohydrate (in form of lactose)	6.5 to 7.5 percent	4.5 to 5.0 percent
Fat(s)	Variable, but both have approximately 3.5 percent. (Differs qualitatively)	
	Contains more olein, which is is readily adsorbed	Contains more volatile fatty acids, which are irritating to the gastric mucosa
	Digestion of fat easy	Digestion of fat sometimes difficult
Minerals	0.15 to 0.25 percent	0.7 to 0.75 percent. Contains more of all minerals with the exception of iron and copper
	Iron content is low in both milks, approximately:	
	1.5 mg/1	0.5 mg/1
Vitamins	Varies with maternal intake	

(continued)

Table 1 (cont'd.)

Parameter	Human Milk	Cow's Milk
Vitamin A	Relative large amounts in both milks	
Vitamin B	Probably adequate in both milks	
Vitamin C	More is found in human milk	
Thiamine	Higher content in cow's milk	
Riboflavin	Higher content in cow's milk	
Vitamin D	Relatively small amount in both milks	
Vitamin E	Satisfactory level in breath milk	
Digestion	Cow's milk has a higher buffer content and can therefore adsorb much more gastric acid than breast milk before it reaches the acidity necessary for digestion. The large amount of casein on cow's milk make large, tough curds in the stomach as compared with the fine, easily broken down curds of breast milk	

The purpose of this study was to measure levels of environmental pollutants in human milk by gas chromatography/mass spectrometry (GC/MS) and to evaluate the utility of using this body fluid in specific pollutant studies for populations in the vicinity of chemical manufacturing plants and/or industrial user facilities. All routes of exposure, i.e., air, water, particulate, clothing and food were of interest. Mother's milk samples were acquired and analyzed for selected industrial chemicals. The chemicals of interest included: polychlorinated naphthalenes (PCNs), tetrachloroethylene, trichloroethane, dichloropropanes, benzene, polybrominated biphenyls (PBBs), chlorinated phenols, toluene, chlorinated benzenes, and chloroform.

Where possible, any other chemicals found in the extracts were identified and quantitated. The levels of selected organic compounds in mother's milk were investigated to assess the possibility of using this medium as an indicator of body burden for a wide range of organic compounds. For this feasibility study, no attempts were made to develop a statistically valid sample; sites were selected as having a high probability of pollutant detection and subjects were selected on a volunteer basis.

LITERATURE REVIEW

A review of the literature concerning pollutants in mother's milk was conducted. A computer search of MEDLARS II and ORBIT--III yielded 108 citations. These citations, plus personal contacts and manual searches yielded the data discussed below.

By far, most of the literature on environmental pollutants in mother's milk deals with chlorinated insecticides (e.g. DDT). PCBs have also been studied. Only a few references discuss the presence of other compounds in milk.

Table 2 lists the levels of pollutants found in mother's milk in the United States. Table 3 summarizes these findings. Table 4 summarizes pollutants found in mother's milk outside the United States. With the exception of one reference(27) regarding 1,2-dichloroethane exposure, all of the compounds found in mother's milk are semivolatile (extractable) halogenated compounds.

Table 2. LEVELS OF ORGANIC COMPOUNDS FOUND IN HUMAN MILK IN THE UNITED STATES

Compound	Sample Matrix	Mean (ppb)	Range (ppb)	Number of Determinations	Locations	References
β-BHC	Milk	0.5	T-10	57	AR, MS	2
	Milk		T-28	40	CO	3
γ-BHC	Milk Fat	83	30-270	53	PA	4
Total BHC	Milk	6.5	<0.1-20.2	14†	US	5
	Milk	7.7	n.d.-37.0	28	TX	6
	Milk	6.2	3.6-9.0	7	Houston, TX	6
p,*p*'-DDD	Milk	4.7	<0.1-14	14†	US	5
	Milk Fat	10.8	n.d.-30	53	PA	4
	Milk		T-5	40	CO	3
o,*p*'-DDE	Milk	1.0	<0.1-2.8	14†	US	5
p,*p*'-DDE	Milk	227	10-1720	57	AS, MS	2
	Milk	29	5.2-981	14†	US	5
	Milk	84.1	13.4-236	28	TX	5
	Milk	92.4	16.7-138	7	Houston, TX	6
	Milk Fat	1766	790-4350	53	PA	4
	Milk		79-386	40	CO	3
DDE	Milk	194	74-314	30*	AZ	7
	Milk	60	20-90	4	Chicago, IL	8
	Milk	30	<10-140	5	Wenatche, WA	8
	Milk	30	-**	1**	Phoenix, AZ	8
	Milk	100	70-120		US	8

(continued)

Table 2 (cont'd.)

Compound	Sample Matrix	Mean (ppb)	Range (ppb)	Number of Determinations	Locations	References
o,p'-DDT	Milk	92	10-840	57	AR, MS	2
	Milk	25	<0.1-10.8	14†	US	5
	Milk	10	5-36	30*	AZ	7
	Milk		T-13	40	CO	3
p,p'-DDT	Milk	29	7.8-89	14†	US	5
	Milk	114	9-383	30*	AZ	7
	Milk Fat	513	90-2120	53	PA	4
	Milk		7-109	40	CO	3
DDT (unspecified)	Milk	100	80-130	4	Chicago, IL	8
	Milk	60	<10-220	5	Wenatche, WA	8
	Milk	60	-**	1	Phoenix, AZ	8
	Milk	70	50-90	**	US	8
	Milk		10-110	40	CO	3
	Milk	130	n.d.-770	32	DC	9
Total DDT Equiv.	Milk	334	20-2760	57	AR, MS	2
	Milk	70.5	40.4-156	14	US	
	Milk	100	SD=100	14	Long Island, NY	10
	Milk	170	SD=130	20	Rochester, NY	10
	Milk	180	SD=100	19	Chicago, IL	10
	Milk	220	SD=170	27	Lexington, KY	10
	Milk	170	SD=150	34	Nashville, TN	10
	Milk	150	SD=80	6	Memphis, TN	10
	Milk	180	SD=120	18	Los Angeles, CA	11
	Milk	447	59-1899	38	MS, AK	11
	Milk	75	15-133	14	Nashville, TN	11
	Milk	323	185-721	7	MS, AK	11
	Milk	130	n.d.-770	32	Washington, DC	9

(continued)

Table 2 (cont'd.)

Compound	Sample Matrix	Mean (ppb)	Range (ppb)	Number of Determinations	Locations	References
Dieldrin	Milk	0.4	T-50	57	AR, MS	2
	Milk	6.2	2.9-14.6	14†	US	5
	Milk	3.3	n.d,-21	28	TX	5
	Milk	7.5	1.9-21	7	Houston, TX	5
	Milk		T-11	40	CO	3
Heptachlor Epoxide	Milk	4	T-30	57	AR, MS	2
	Milk	1.7	<0.1-4.4	14†	US	5
	Milk Fat	160	40-460	53	PA	4
	Milk		T-5	40	CO	3
t-Nonachlor	Milk	1	T-10	57	AR, MS	2
Oxychlordane	Milk	5	T-20	57	AR, MS	2
PCBs	Milk	T	T	57	AR, MS	2
	Milk	~10	<40-100	39	CO	12
	Milk		40-100	40	CO	3
Nicotine	Breast Fluid		n.d.-195	6	CA	13

NOTES: BHC = benzenehexachloride (hexachlorocyclohexane)
DDD = 2,2-bis(chlorophenyl)-1,1-dichloroethane
DDE = 1,1-dichloro-2,2-bis(chlorophenyl)ethylene
DDT = 1,1,1-trichloro-2,2-bis(chlorophenyl)ethane
Total DDT equiv. = sum of all DDT-related peaks calculated as if all were DDT
PCBs = polychlorinated biphenyls. Quantitation generally based on comparison to an Aroclor mixture
T = trace
n.d. = not detected
SD = standard deviation
† = 5 women. Separate determinations make total of 14 samples.
* = 6 women. Separate samples makes total of 30 samples.
** = unspecified pool of donors in Denver and other US areas, no range given.
Missing values indicate no data in original article

Table 2 (cont'd.)

NOTES (cont'd.): Mean values were taken from original citation where available; otherwise arithmetic mean was calculated, counting "ND" values as zero and "T" values as 0.5 times the lowest reported value.

Table 3. RANKING OF PESTICIDES AND PCBs BY REPORTED CONCENTRATIONS IN HUMAN MILK[a]

Compound	Weighted Mean Concentration (ppb)[b]	Number of Samples
DDE[c]	99	103
DDT[c]	94	100
PCBs[c]	<10	96
Oxychlordane	5	57
Dieldrin	4	92
DDD[c]	4	54
Heptachlor epoxide	4	71
BHC[c]	3	106
t-Nonachlor	1	57

[a] Whole milk only.

[b] Mean value calculated from a weighted mean of values in Table 2. Where either the mean or number of samples analyzed were unavailable, the data were excluded from calculation.

[c] All isomers summed.

Table 4. LEVELS OF ORGANIC COMPOUNDS FOUND IN HUMAN MILK OUTSIDE THE UNITED STATES

Compound	Sample Matrix	Mean (ppb)	Range (ppb)	Number of Determinations	Number of Positives	Location	Date	Reference
α-BHC	Milk	0.58	0.1-1.9	50	17	Norway	1975	14
β-BHC	Milk	4.69	1.2-17.8	50	49	Norway	1975	14
	Milk	70	ND-900	96	64	Germany	1971	15
	Milk	200	80-910	22	19	Vienna	1973	16
	Milk	280	10-850	9	7	Rural Austria	1973	16
	Milk	4	1-16	50	42	Leiden (Neth.)	1969	17
	Milk	2	ND-21	100	91	Canada	1975	13
γ-BHC	Milk	10.91	1.0-35.8	50	17	Norway	1975	14
	Milk	-	ND	96	0	Germany	1971	15
	Milk Fat	48	26-114	22	19	Vienna	1973	16
	Milk Fat	63	40-100	9	7	Rural Austria	1973	16
	Milk	10.1		29		Israel	1975	18
	Milk	3	<1-35	147		Canada	1967-8	19
δ-BHC	Milk	1.14	0.3-3.2	50	34	Norway	1975	14
Total BHC	Milk	9.4	1.7-45.5	50	50	Norway	1975	14
	Milk	13	7-33	19	19	England	1964	20
p,p'-DDD	Milk	9.9		29		Israel		18
DDD	Milk	7	3-14	67	12	Australia	1970	21
o,p'-DDE	Milk	18.02	1.6-43.8	50	30	Norway	1975	14
	Milk	9.5		29		Israel	1975	18
p,p'-DDE	Milk	65.10	0.9-113.2	50	50	Norway	1975	14
	Milk		6-699	168	167	Portugal	1972	22
	Milk	90	ND-600	96	95	Germany	1971	15
	Milk	21.7		29		Israel	1975	18
	Milk	97	6-770	147		Canada	1967-8	19
	Milk	30		50	50	Leiden (Neth.)	1969	17
	Milk	35	17-68	6	6	New Brunswick	1973	23
	Milk	19	9-40	9		Nova Scotia	1973	23
	Milk	35	7-144[a]	100	100	Canada	1975	24
	Milk	73	40-100	19	19	England	1964	20

(continued)

Table 4 (cont'd.)

Compound	Sample Matrix	Mean (ppb)	Range (ppb)	Number of Determinations	Number of Positives	Location	Date	Reference
DDE	Milk	105	12-450	67	67	Australia	1970	21
	Milk Fat	3380	1930-7950	22	22	Vienna	1973	16
	Milk Fat	3920	3420-5970	9	9	Rural Austria	1973	16
	Milk	61	15-112	26	26	W. Australia	1970-1	25
o,p'-DDT	Milk	18.52	1.6-120.9	50	49	Norway	1975	14
	Milk	7.3		28		Israel	1975	18
	Milk	5	<1-31	147		Canada	1967-8	19
	Milk	3	ND-48	100	32	Canada	1975	24
p,p'-DDT	Milk	17.89	2.3-138.3	50	50	Norway	1975	14
	Milk		3-345	168	167	Portugal	1972	22
	Milk	90	10-250	96	95	Germany	1971	15
	Milk	7.3		29		Israel	1975	18
	Milk	32	3-344	147		Canada	1967-8	19
	Milk	16		50	50	Leiden (Neth.)	1969	17
	Milk	13	6-30	6	6	New Brunswick	1973	23
	Milk	6	<2-11	9	9	Nova Scotia	1973	23
	Milk	6	7-21[a]	100	100	Canada	1975	24
	Milk	45	20-75	19	19	England	1964	20
DDT	Milk	36	7-160	67	67	Australia	1970	21
	Milk Fat	1060	300-2680	22	21	Vienna	1973	16
	Milk Fat	1760	1030-2530	9	9	Rural Austria	1973	16
	Milk	10	2-25	26	26	W. Australia	1970-1	25

(continued)

Table 4 (cont'd.)

Compound	Sample Matrix	Mean (ppb)	Range (ppb)	Number of Determinations	Number of Positives	Location	Date	Reference
Total DDT Equiv.	Milk	81.74	5.2-349.0	50	50	Norway	1975	14
	Milk	186	<10-780	160	167	Portugal	1972	22
	Milk Fat	1390	220-2580	19	19	Ontario	1973-4	26
	Milk Fat	3480	330-18800	34	34	Ontario	1971-2	26
	Milk Fat	3480	110-11400	48	48	Ontario	1969-70	26
	Milk	320	30-870	96	96	Germany	1971	15
	Milk	141	15-580	67	67	Australia	1970	21
	Milk	139	10-1020	147		Canada	1967-8	19
	Milk	78	19-137	26	26	W. Australia	1970-1	25
	Milk	378	3-5868	290	290	Guatemala	1973-4	27
	Milk	128	75-170	19	19	England	1964	24
Dieldrin	Milk	2.75	0.3-3.6	50	6	Norway	1975	14
	Milk	40	5-31	168	15	Portugal	1972	14
	Milk Fat	40	<10-80	19		Ontario	1973-4	26
	Milk Fat	90	<10-170	34		Ontario	1971-2	26
	Milk Fat	90	<10-250	48		Ontario	1969-70	25
	Milk	6	1-29	67	29	Australia	1970	21
	Milk	7.0		29		Israel	1975	18
	Milk	5	1-60	147		Canada	1967-8	19
	Milk	5	3-11	26	26	W. Australia	1970-1	25
	Milk	3	0.1-10.7	50	48	Leiden (Neth.)	1969	17
	Milk	2	ND-6	100	84	Canada	1975	24
	Milk	6	1-13	19	19	England	1969	20
Aldrin	Milk	21.8		50	1	Norway	1975	14
Heptachlor Epoxide	Milk	1.57	0.6-2.6	50	18	Norway	1975	14
	Milk	9.1		29		Israel	1975	18
	Milk	3	<1-23	147		Canada	1967-8	19
	Milk	1.2	0.3-3.5	50	50	Leiden (Neth.)	1969	17
	Milk	1	ND-3	100	69	Canada	1975	24

(continued)

Table 4 (cont'd.)

Compound	Sample Matrix	Mean (ppb)	Range (ppb)	Number of Determinations	Number of Positives	Location	Date	Reference
HCB	Milk	9.1	1.7-60.5	50	50	Norway	1975	14
	Milk Fat	100	ND-250	19		Ontario	1973-4	26
	Milk Fat	1240	260-4360	22	22	Vienna	1973	16
	Milk	3670	2140-5110	9	9	Rural Austria	1973	16
	Milk	25	12-34	26	26	W. Australia	1970-1	25
	Milk	2	ND-21	100	81	Canada	1975	24
PCB	Milk Fat	1200	100-2500	19	19	Ontario	1973-4	26
	Milk Fat	1200	200-3000	34	34	Ontario	1971-2	26
	Milk Fat	1000	700-12000	48	48	Ontario	1969-70	26
	Milk	90		96	64	Germany	1971	15
	Milk Fat	1540	580-3780	22	22	Vienna	1973	16
	Milk Fat	1290	950-1570	9	9		1973	16
	Milk	22	15-30	6	6	New Brunswick	1973	23
	Milk	18	12-32	9	9	Nova Scotia	1973	23
	Milk	12	ND-68	100	100	Canada	1975	24
Oxychlordane	Milk	1	ND-2	100	77	Canada	1975	24
trans-Nonachlor	Milk	1	ND-2	100	77	Canada	1975	24
1,2-Dichloroethane	Milk	6000		1	1			28

NOTES:

BHC = benzenehexachloride (hexachlorocyclohexane)
DDD = 2,2-bis(chlorophenyl)-1,1-dichloroethane
DDE = 1,1-dichloro-2,2-bis(chlorophenyl)ethylene
DDT = 1,1,1-trichloro-2,2-bis(chlorophenyl)ethane
Total DDT equiv. = sum of all DDT-related peaks calculated as if all were DDT.
PCB = polychlorinated biphenyls. Quantitation generally based on comparison to an Aroclor mixture.

HCB = hexachlorobenzene
ND = not detected.

Mean values were taken from original citation where available; otherwise arithmetic mean was calculated, counting "ND" values as zero and "T" values as 0.5 times the lowest reported value.

Missing values indicate no data in original article.

[a] Lowest value not reported.

The literature shows that mother's milk often contains semivolatile chlorinated organic pollutants (pesticides). Presumably due to lack of analytical techniques and/or sensitivity, the presence of other pollutants has apparently not been investigated.

SECTION 2

SUMMARY AND CONCLUSIONS

The results show that sampling and analysis for organic compounds in mother's milk is feasible. The sample collection technique presented no significant problems. Analysis of the samples was generally satisfactory.

The use of purge and trap with gas chromatography/mass spectrometry/computer (GC/MS/COMP) analysis for volatile organics was successful, although the intrusion of contaminants during analysis presented problems with some compounds. The wide range of volatile compounds found includes common air and water pollutants and possible metabolites. Thus, it may be possible to use mother's milk as an indicator of body burden if a correlation between exposure and mother's milk concentration is established.

The extraction and GC/MS analysis for semivolatile organics was only marginally successful due to limited sensitivity (about 20-100 ppb milk). PCBs and DDE were the only halogenated semivolatiles found. The target semivolatile compounds (PCNs, PBBs, chlorinated phenols, and the higher chlorinated benzenes) were not present in quantities detectable by the survey techniques. The use of more sensitive (generally a factor of 100-1000) and selective methods [GC/electron capture detection (ECD), GC/negative ion chemical ionization mass spectrometry (NICIMS) or GC/single ion monitoring MS] may detect these compounds, but was outside the scope of this project.

SECTION 3

RECOMMENDATIONS

Further studies of the applicability of mother's milk as a matrix for assessing the human body burden of pollutants must directly compare human milk with the other available sample matrices. For example, comparison of the volatiles in breath, blood, urine, and mother's milk would determine which matrices are most suitable for measuring these compounds. It may also be advisable to use animal studies to determine the extent of environmental exposure-body burden correlation.

In addition, the effects of transport of pollutants to a newborn infant should be studied. Infants may be uniquely affected by some pollutants due to their small body weight and different metabolism relative to adults.

The measurement of semivolatile organics in mother's milk requires more sensitive techniques than those used in this study. For example, chlorinated compounds could best be detected using GC/ECD or GC/negative ion chemical ionization mass spectrometry and polynuclear aromatics by GC/photoionization detection.

Improvement in analytical methodology could occur at several points:

(1) As discussed above, more sensitive, analytical procedures could be used for specific compound classes.

(2) For volatile organics, background levels could be reduced with an on-line purge and trap/GC system.

Potential improvements in survey and sampling methodology include:

(1) Addition of questions regarding length of nursing, age of infant, time since last nursing, etc.

(2) Selection of participants according to a more statistically valid method (e.g. statistically random sampling).

(3) Closer control over physical collection methodologies (e.g. all respondents gathered at one location).

The 5-month time lag in the study awaiting OMB clearance was seriously detrimental to the project. The personnel and apparatus used for the validation studies had to be reassembled once OMB clearance was obtained. Restarting a project following a long dormant period requires retraining analytical personnel (or training new personnel if original personnel have been reassigned to other research projects), recalibration of instruments, and assembling the necessary laboratory apparatus and supplies, all of which consume government resources. Reducing this time lag is extremely important for execution of programs involving human testing.

SECTION 4

SELECTION OF SAMPLING SITES

Five urban areas were chosen as sampling sites. Each of these cities is a high-probability area for the presence of one or more of the chemicals of interest in mother's milk. Since many of the compounds of interest are probably specific to certain industrial sites, the samples from the other sites were intended to serve as controls for the site-specific compounds. Other compounds are considered ubiquitous and their levels in milk was probably not related to local industrial activity. The rationale for selecting the five sampling sites is discussed below.

BRIDGEVILLE, PENNSYLVANIA

PCNs are manufactured by Koppers Company, Inc., of Pittsburgh, PA, at the Koppers Chemical and Coatings plant in Bridgeville, about 10 km SW of Pittsburgh.(29) Reported production levels were 7 million lb in 1956 and 5 million lb in 1972,(29) indicating a potential long-term, relatively constant, exposure level in the surrounding area. Results from environmental monitoring in the area immediately (< 1 km) surrounding the plant indicated higher levels of PCNs in air and soil than those found near five PCN user sites, as shown in Table 5.(30-34) Furthermore, fish and apple samples from the same area were found to contain PCNs, indicating a potential link to the human food chain.

In addition to PCNs, plants in the Bridgeville area have been reported to emit large quantities of phthalic anhydride particulate.(35) At this plant site, Koppers is reported to manufacture chlorinated naphthalenes, phthalic anhydride, maleic anhydride, and alkyd resins.(36)

Table 5. SUMMARY OF PCN CONCENTRATIONS FOUND NEAR MANUFACTURING AND USE SITES(32)

Site	Sampling Period	Air, ng/m^3			Water, μg/L		Soil, μg/kg		
		Low	High	Mean	Up-stream	Down-stream	Low	High	Mean
PCN manufacturer (Koppers)	1	25	450	150	0.2	1.4	130	2300	940
	2	120	2900	1400	a				
Capacitor manufacturing A	1	ND[b]	7.3	3.1	ND	ND	ND	7.3	2.0
	2	ND	3.9	1.2					
Capacitor manufacturing B	1	9.8	31	19	ND	0.6	ND	470	100
	2	9.8	33	17					

[a] No water samples collected for period 2.

[b] Not detected.

NORTHERN NEW JERSEY - STATEN ISLAND, NEW YORK, AREA (NNJ)

The Northern New Jersey (NNJ) area was selected as a sampling site on two bases: production of PBBs and general chemical industrial activity.

Three facilities are of interest(37) with respect to PBBs: White Chemical Co., E 22nd St., Bayonne, NJ; Marcor, Inc., Standard T. Chemical Co., subsidiary, 2500 Richmond Terrace, Staten Island, NY; and Hexcel Corp., Fine Organics Division, 880 Main St., Sayreville, NJ. White produced 45,000 kg of PBBs (specifically octabromobiphenyl and decabromobiphenyl) between 1970 and 1973.(38) Hexcel is reported(39) to have produced unspecified amounts of decabromobiphenyl [as well as to have produced or used decabromobiphenyl oxide, ethylene dichloride, and 1,2-bis(2,4,6-tribromophenoxy)ethane]. Standard T is thought to have been a PBB user up to about 1974.(39)

Results of environmental sampling in the area surrounding these three companies(40,41) indicated the presence of PBBs, especially the more highly brominated homologs, in sediment, water, soil, human hair, fish, turtle, and plant matter. The findings in human hair oil (18 total samples), which ranged from undetectable to 310 ppm, are especially relevant to this study, since they indicate that the PBB manufacturing in this area and the resultant environmental contamination has resulted in human exposure.

Northern New Jersey has a high concentration of chemical industries,(42) many of which use or produce halogenated hydrocarbons. The list of industries and locations are summarized below. Coastal Industries, Inc. (swimming pool chemicals), Diamond Shamrock (textile processing chemicals), Scientific Chemical Processing (chemical waste disposal) and Tenneco Chemicals (synthetic foam rubbers) are located in Carlstadt. Crompton & Knowles Corp. (dyes, colors and chemicals) are located in Fairlawn. Fisher Scientific (chemicals), Conoco Chemicals are in Saddle Brook. In Bayonne are CIBA-Geigy (dyes and intermediates) and ICI America (organics). In Jersey City are Mallinkrodt (analytical reagents) and Onya Chemical Co. (textile finish compounds, water repellants, germicides, and detergents). In Kearney are Standard Chlorine Chemical Co. (chlorobenzenes), Theobald Industries (bleaches), PPG Industries (paint) and Monsanto (industrial chemicals). In South Kearney is BASF-Wyandotte (dyestuffs and vinylidine chloride). In Newark are American Oil and Supply Co. (surfactants and chemicals), Celanese Plastics (plastics), DuPont (pigments), Inmont (paint), Maas & Waldstein (paint), Otto B. May (dyes, surfactants), 3M (chemicals), Benjamin Moore (paint), Sherwin-Williams (paint) and Vulcan Materials (chloromethanes). In Elizabeth are Perk (chlorinated solvents) and Speciality Chemicals Division of Allied Chemical Corp. Linden Chlorine Products (chlorine) is in Linden. In Rahway are M & T Chemicals (speciality chemicals) and Merck and Co. (industrial chemicals). In Edison are Cary Page Chemicals (PVC compounds) and Mobile Chemical (paint). In Parlin, Hercules manufactures chloroform. In Passaic are Pantasote Co. of New York (PVC resin film), Stauffer (vinyl sheet and film) and United Wool Piece Dyeing and Finishing (dyes). In Patterson are several dye manufacturers. In Wayne are American Cyanamid (chemicals) and Owens Illinois (plastics). Many of these and other firms in NNJ undoubtedly manufacture or use compounds which are of interest to this study.

The levels of general organic pollutants in NNJ have been found to be high due to intense chemical manufacturing in the area. Environmental monitoring by RTI under separate contracts,(43-46) has found a wide variety of organic pollutants in this area. In addition, preliminary results from ground and surface water samples indicate measurable levels of a number of volatile halogenated hydrocarbons.(44,45) These data, summarized in Table 6, are indicative of environmental levels of organics in the NNJ area to which humans may be exposed and thus are indicative of the types of compounds anticipated in mother's milk. Under a separate research project,(45) the daily intake of some selected organics was roughly estimated. These estimates are given in Tables 7 and 8. Clearly there is ample exposure to pollutants which could potentially partition into milk.

The statistics for cancer in two counties of NNJ are very high.(58,59) The overall rate for all malignant neoplasms is significantly above the national average. This cancer incidence in New Jersey has been partially linked to the chemical and allied industries located there.(60-64)

Northern New Jersey is a metropolitan area with a relatively static population, a well-established chemical industry, known environmental levels of organics (including PBBs) and abnormally high cancer rates. These factors make this area especially suited to this study of organics in mother's milk.

Table 6. PREVALENT HALOGENATED COMPOUNDS IN AMBIENT AIR AND WATER OF RAHWAY/WOODBRIDGE, BOUNDBROOK AND PASSAIC, NJ(44)

	Occurrence			
Medium	Ubiquitous	Mean Concentration[a]	Area Specific	Mean Concentration[a]
Air	tetrachloroethylene	210,000	1,1,2-trichloroethane	9,000
	trichloroethylene	125,000	vinyl chloride	1,200
	1,1,1-trichloroethane	62,000	1,2-dichloroethylene	1,000
	1,2-dichloroethane	96,000	1,1,2,2-tetrachloroethane	750
	chloroform	47,000		
	carbon tetrachloride	29,000		
	o,*m*,*p*-dichlorobenzenes	11,000		
	chlorobenzene	2,700		
Water	dichlorobenzene	209	chloronitrobenzene	10.7
	trichloroethane	42	methyl trichlorophenoxy acetate	5
	chloroform	14	methyl dichlorophenoxy acetate	3.5
	trichloroethylene	7	bromopropylbenzene	3
	dichloroethane	5	bromobenzene	3
	bromodichloroethane	5	tetrachloroethane	2.5
	bromodichloromethane	3.7	dichloroethylene	1.8
	tetrachloroethylene	3.6		
	dibromochloromethane	3.3		

[a] Concentrations for air expressed in ng/m^3 and for water in μg/L.

Table 7. ESTIMATED DAILY INTAKE OF SELECTED VOLATILE COMPOUNDS AND EXPECTED CONCENTRATIONS IN BLOOD IN NORTHERN NEW JERSEY(45)

Toxic Chemical	Air[a] (ng/day)	Water[b] (ng/day)	Food[c] (ng/day)	Total (ng/day)	Potential Blood Concentration[d] (ppb)
tetrachloroethylene	2,100,000	3,600	4,150	2,108,000	88
trichloroethylene	1,250,000	7,000	18,660	1,276,000	53
1,1,1-trichloroethane	620,000	42,000	5,290	667,000	28
1,2-dichloroethane	960,000	5,000		965,000	40
chloroform	470,000	14,500	14,280	499,000	21
carbon tetrachloride	290,000	1,000	12,070	303,000	13
dichlorobenzene	110,000	209,000		319,000	13
chlorobenzene	27,000	1,000		28,000	1.2
vinyl chloride	12,000			12,000	0.5
bromodichloromethane		3,700		3,700	0.2
benzene	7,500[e]	300[f]		7,800	0.2
total				6,188,200	258.2

[a] From Ref. 44, calculated on basis of 10,000 L/24 h respiration rate.

[b] From Ref. 44, calculated on basis of 1 L/24 h intake.

[c] From Ref. 47, calculated from FDA standard diet (Ref. 48).

[d] Expected blood concentration is total daily intake divided by blood volume (8.000 mL) assuming 4 half-lives/day.

[e] From Ref. 49, 50.

[f] From Ref. 50.

Table 8. TOTAL DAILY INTAKE OF TARGET COMPOUNDS, PESTICIDES, PCBs, BaP AND METALS AND CONCENTRATIONS IN BLOOD IN NORTHERN NEW JERSEY(45)

Toxic Chemicals	Air (ng/day)	Water (ng/day)	Food (ng/day)	Total (ng/day)	Expected Blood Concentration (ppb)
α-BHC	10	0	1,100	1,110	0.14
lindane	60		586	646	0.08
heptachlor	30	0	62	92	0.01
heptachlor epoxide		7	640	647	0.08
chlordane	20			20	~0
DDE			3,500	3,500	0.44
DDT/DDD	70	0	2,500	2,570	0.32
HCB	50		73	123	0.02
PCBs	~200	<60	388	648	0.08
Total Halogenated Compounds	440	<67	8,849	9,356	1.16
benzo(a)pyrene	21	2	7,800	7,823	1.0
arsenic	2,800	<1,000	31,300	34,100	4.4
cadmium	50	<1,000	32,000	33,000	<10[a]
lead	7,500	3,200	105,000	115,700	100-500[b]

[a] Ref. 56.

[b] Ref. 57.

Table 8 (cont'd.)

Sources:

Pesticides and PCBs in air -- Ref. 51 (US)
Pesticides in water -- Ref. 44 (NJ)
Pesticides and PCBs in food -- Ref. 48 (US)
PCBs in water -- Ref. 51 (US)
BaP in air -- Ref. 52 (US)
BaP in water -- Ref. 53 (World)
BaP in food -- Rough estimation (from Ref. 53 [World])
Metals in air -- Ref. 54 (NJ)
Metals in water -- Ref. 55 (NJ)
Metals in food -- Ref. 48 (N.E. NJ)

BATON ROUGE, LOUISIANA

Baton Rouge was selected on the basis of extensive organic chemical production (especially volatile halogenated hydrocarbons) as summarized in Table 9.(43) In addition, RTI has collected and analyzed ambient air samples from this area and established the presence of a number of compounds of interest in ambient air.(43) A summary of the levels of halogenated compounds found in water and air is presented in Table 10.

In addition to the industrial production in Baton Rouge, industries in Plaquemine (15 km SSW), St. Gabriel (20 km SSE) and Geismar (27 km SSE) may emit significant levels of chemicals which may contribute to the levels observed in mother's milk in Baton Rouge. These industries and their production are listed in Table 11.(36)

KANAWHA VALLEY, WEST VIRGINIA

Many manufacturers of organic chemicals are located in the Kanawha Valley, WV. DuPont, near Belle, WV, has a large chemical complex for the synthesis of substances such as methylmethacrylate, methylamines, ammonia, hydrogen cyanide, herbicides, and insecticides. In South Charleston are production and consumption plants (Union Carbide, and FMC). Plastics, PVC, antifreeze, chlorine, halogenated organics, carbon disulfide, peroxides, etc., are the predominant chemicals produced here. The major industrial facility in the town of Institute is Union Carbide, which also processes a broad spectrum of compounds, e.g., viscose rayon and phthalate esters. There is also a large-scale olefin processing complex and a rubber accelerator plant. A major terminal loading facility in South Charleston handles large quantities of a variety of organic compounds. Monsanto, FMC, Allied, and Fike have plants near Nitro for the production of antioxidants, rubber accelerators, industrial chemicals, and other materials. Several other chemical manufacturers, consumers, and transporters are located in the Kanawha Valley, some or all of which may contribute to the presence of organic materials in the ambient air or water and thus contribute to human exposure.

Previous RTI sampling(43,46,65,66) in the Kanawha Valley found a broad range of halogenated, ketone, aldehyde, ester, aromatic, and aliphatic compounds. Quantitative results included high values in air of 11,000 ng/m^3

Table 9. POTENTIAL EMISSIONS FROM CHEMICAL INDUSTRY IN BATON ROUGE, LA[a](43)

Chemical	Total Production (mmlb/yr)	Raw Material	Company[b]
chlorodifluoromethane (101)	-	chloroform	ACC[c]
dichlorodifluoromethane (12)	-	carbon tetrachloride	ACC
dichlorotetrafluoroethane (114)	NA	perchloroethylene	ACC
ethylene dichloride	1100	ethylene	ACC, EC
polyethylene resin	460	ethylene	ACC
trichlorofluoromethane (11)	-	-	ACC
1,1,2-trichloro-1,2,2-trifluoroethane (113)	NA	perchloroethylene	ACC
vinyl chloride	480	ethylene dichloride	ACC, EC
ethyl chloride	210	ethylene	EC
methyl chloride	75	methanol	EC
perchloroethylene	100	ethylene dichloride	EC
tetraethyl lead	312	ethyl chloride	EC
1,1,1-trichloroethane	40	1,1-dichloroethane	EC
trichloroethylene	32	ethylene	EC
PVC	144	-	EC
benzene	440	petroleum	EXCC
butadiene	428	ethane, etc.	EXCC, CRCC
n-butyl alcohol	NA	-	EXCC

(continued)

Table 9 (cont'd.)

Chemical	Total Production (mmlb/yr)	Raw Material	Company[b]
decanol[c]	NA	nonene	EXCC
diisodecylphthalate	NA	phthalic anhydride, isodecanol	EXCC
dodecene	100	propane/propylene	EXCC
ethylene	700	ethane, etc.	EXCC
isobutylene	NA	petroleum	EXCC
isodecanol[c]	NA	nonene	EXCC
isooctyl alcohol[c]	NA	neptene	EXCC
isoprene	10	ethylene by-product	EXCC
isopropanol	680	propylene	EXCC
neopentanoic acid	5.5	isobutylene	EXCC
nonene	300	propane/propylene	EXCC
phthalic anhydride	90	o-xylene	EXCC
propylene resin	320	ethylene	EXCC
toluene	378	petroleum	EXCC, FGC
ethylbenzene	900	benzene	FGC
styrene	800	ethylbenzene	FGC
vinyl toluene	NA	toluene, ethylene	FGC

[a] Data provided by the Louisiana State Air Board.

[b] ACC = Allied Chemical Corp., EC = Ethyl Corp., EXCC = Exxon Chem. Corp., FGC = Foster-Grant Co. Inc.

[c] Involves production of other alcohols also, C_6, C_8, C_9, C_{10}, C_{13}, C_{16}.

NA = not available.

Table 10. PREVALENT HALOGENATED COMPOUNDS OCCURRING IN AMBIENT AIR AND WATER OF BATON ROUGE, GEISMAR AND PLAQUEMINE, LA(44)

Medium	Occurrence			
	Ubiquitous	Mean Concentration[a]	Area Specific	Mean Concentration[a]
Air	chloroform	5,500	1,1,2-trichloroethane	632
	1,2-dichloroethane	1,656	1,2-dichloroethylene	472
	carbon tetrachloride	811	dichlorobutane	409
	1,1,1-trichloroethane	605	1,2-dichloropropane	306
	trichloroethylene	142	vinylidene chloride	78
	tetrachloroethylene	118	1,1,2,2-tetrachloroethane	70
	1,1-dichloroethane	86		
Water	trichloroethylene	96	bromobenzene	13
	chloroform	20	1,2-dichloroethylene	4
	trichloroethane	11	hexachloroethane	1.6
	dichloroethane	7.7		
	carbon tetrachloride	7.1		
	dichlorobenzene	4.2		
	chlorodibromomethane	3.5		
	tetrachloroethylene	1.9		

[a] Concentrations for air expressed in ng/m^3 and for water in µg/L.

Table 11. POTENTIAL EMISSIONS FROM CHEMICAL INDUSTRY IN PLAQUEMINE, GEISMAR, AND ST. GABRIEL, LA(36)

City	Chemical	Annual Capacity (million pounds)	Company[a]
Plaquemine	chloroform	b	Dow
	1,2-dichloropropane	10	"
	ethylene dichloride	1325	"
	methyl chloride	150	"
	methylene chloride	190	"
	tetrachloroethylene	150	"
	vinyl chloride	450	"
Geismar	chloroform	46	VCM
	ethylene dichloride	330	"
	methylene chloride	80	"
	tetrachloroethylene	150	"
	1,1,1-trichloroethane	65	"
	phosgene	55	BASF
	phosgene	125	RCC
	vinyl chloride	300	BOR
	vinyl chloride	300	MCJ
St. Gabriel	phosgene	NA	SCC

[a] Dow = Dow Chem. USA
VMC = Vulcan Materials Co.
BASF = BASF Wyandotte Corp.
RCC = Rubicon Chems., Inc.
BOR = Borden, Inc.
MCI = Monochem, Inc.
SCC = Stauffer Chem Co., Agric. Chem. Div.

[b] 200 million pounds combined capacity in Plaquemine and Freeport, TX plants.

for methylene chloride, 1500 ng/m^3 for tetrachloroethylene, and 72,000 ng/m^3 for benzene. Compounds identified in the air particulate fraction included long-chain alkanes, polycyclic aromatic hydrocarbons (PAH) from naphthalene through anthanthrene (or an isomer), alkyl-PAH derivatives, and nitrogen-containing heterocycles.

SECTION 5

SAMPLE COLLECTION

At each of the five sites, arrangements were made to work through clinical facilities to recruit a suitable panel of respondents. These facilities included the Bayonne Hospital in Bayonne, NJ; the Medical Center Hospital in Jersey City, NJ; Magee-Women's Hospital in Pittsburgh, PA; Charleston Area Medical Center in Charleston, WV; and the East Baton Rouge Parish Health Clinic in Baton Rouge, LA.

Advance arrangements were made through a contact person at each facility. This person was responsible for recruiting a professional member of the facility's staff to serve as the data collector. The data collector was usually a registered, licensed practical, or public health nurse associated with the facility.

Respondents were paid $5 for their assistance in providing a milk sample and completing the survey questionnaire.

The data collection effort is discussed in the following sections.

OMB CLEARANCE

Under the Federal Reports Act, clearance for the study of human subjects must be obtained from the Office of Management and Budget. This clearance was obtained on October 18, 1978. The OMB number is 158-578010. This study was approved with the understanding that: (1) the surveys were conducted as a pretest of the feasibility of information collection procedures; (2) the information collected will not be used to generalize to either local areas or the nation as a whole. These two caveats were invoked since the sample size was small and a nonprobability sampling method (subject selection) was used.

TRAINING

Before data collection began at a site, a training session was held to acquaint the facility contact person and data collector(s) with the survey. The session addressed the study objectives; use of the data collection instruments; administrative instructions; quality control procedures; and instructions for collecting, packing, and shipping milk samples to RTI. The training was conducted by an RTI survey specialist from the Survey Operations Center. A detailed manual and necessary field reporting forms were developed for use in these sessions. All training was conducted at the participating facility and lasted approximately 4 hours.

SURVEY INSTRUMENTS

Three data collection instruments (see Appendix A) were developed for use by the data collectors. The Participant Consent Form (PCF) was used to introduce the study, explain the study objectives and requirements of participation, present the confidentiality procedures, and obtain consent of participant. This form was signed by the respondent, who retained a copy for her files. The original was attached to the data collection instrument and a second copy was filed in the respondent's hospital record.

The Participant Listing Form (PLF) provided a means of assigning unique numbers to participants at each performance site. The data collector completed this form as each participant was solicited; the form was returned to RTI with the completed questionnaires when work at the site was finished.

The Study Questionnaire (SQ) was the primary data collection instrument. Information concerning participant demographic characteristics, residence information, health data, use of medications, and personal characteristics was obtained through this document. The SQ was administered after patients had been screened and prior to collection of the milk sample.

PARTICIPANT SCREENING

Potential participants (lactating women) were screened by the data collector to determine whether or not they met certain study criteria, which included:

- ability and desire to provide a milk sample of approximately 100 mL.
- permanent residence within the area of interest for at least the preceding 12 months, and
- no travel outside the area of interest for the seven days preceding sample collection.

After potential participants were screened, 10 women who met all the criteria for participation were asked to provide a milk sample and complete the SQ.

PLF, PCF, AND SQ COMPLETION PROCEDURES

When an eligible person agreed to participate, her name was listed on the PLF and she was assigned a unique participant number. The data collector then read the information contained on the PCF to the participant while she followed along using a second copy. After answering questions or handling problems, the data collector asked the participant to sign the PCF prior to administration of the SQ.

The data collector then completed the SQ by asking the questions directly to the participant. Completion time averaged 15 minutes. An adhesive, computer-generated ID label was affixed to the SQ; a duplicate label was provided to be used for identifying the milk sample bottle.

Each participant was a self-respondent unless she was under 18 years of age, in which case the SQ could have been administered in whole or part to the parent or guardian, but in the participant's presence.

SAMPLE COLLECTION PROCEDURES

After completion of the SQ, the data collector made the necessary arrangements for the participant to provide the milk sample. A collection bottle was taken from the shipping box and the adhesive ID label was affixed to the bottle. The milk was manually expressed directly into the bottle; no breast pumps or other devices were allowed. Immediately after the milk was collected, the bottle was capped and the sample frozen until all ten samples were collected and ready for shipment to RTI. A minimum of 60 mL (half-full bottle) was required for each sample. If insufficient milk was collected, the sample was discarded and an additional subject was added to the study.

SHIPPING PROCEDURES

Sample bottles were packed in the shipping container, cooled with dry ice, and sent directly to RTI via Federal Express.

SECTION 6
SAMPLE ANALYSIS METHODS

The milk samples were analyzed using gas chromatography/mass spectrometry/computer. Due to the broad range of volatilities, the samples were partitioned into two general classes of compounds: volatiles (e.g. benzene, chloroform) and semivolatiles (e.g. PCNs, PCBs, pesticides). The analytical protocols developed for the volatile and semivolatile components in mother's milk are reproduced in Appendices B and C, respectively. The experiments conducted which led to these protocols are discussed below.

DEVELOPMENT OF ANALYTICAL PROTOCOL FOR VOLATILES

The headspace purge technique was validated by determining the recovery of four model compounds from raw cow's milk samples. Compounds labeled with carbon-14 were chosen in order to examine both the amounts recovered on Tenax GC and the amounts remaining in purged samples.

Twelve 50 mL cow's milk samples were spiked with methanol solutions of the ^{14}C-compounds. The analysis for each of the four model compounds was performed in triplicate. In addition, standards were prepared in triplicate by adding the appropriate amount of each compound in solution to a scintillation-counting vial containing 15 mL of Triton X/toluene/Omnifluor scintillation "cocktail." Milk samples were purged as described in Appendix B; Tenax cartridges were stored, and aliquots of the purged samples were retained for oxidation and counting.

Tenax cartridges were desorbed at 270°C and 30 mL/min N_2 for 10 minutes into 15 mL of Triton X cocktail in tandem scintillation vials. The vials were capped and refrigerated until scintillation counting. An aliquot (1 mL) of each purged milk sample was oxidized in the Packard Tricarb Sample Oxidizer, which converted all carbon-containing compounds to carbon dioxide and water. The ^{14}C-carbon dioxide was collected in a trapping solution and referenced to a quench correction curve. All standards, Tenax samples and oxidized milk samples were counted on a Packard Liquid Scintillation Counter with automatic standardization. Counting data was analyzed by computer to obtain the number of disintegrations per minute (dpm) for each vial. The percent recovery was calculated for each milk sample as shown below:

$$\text{\% recovery} = \frac{\text{dpm in first vial + dpm in second vial}}{\text{average dpm added to triplicate standards}} \times 100\%$$

The second of the tandem scintillation vials contained <2 percent of the radioactivity in every case. The amounts of ^{14}C compounds retained in the purged sample was calculated:

$$\text{\% retained} = \frac{\text{dpm in oxidized, purged sample}}{\text{average dpm added to triplicate standards}} \times 100\%$$

The data are tabulated in Table 12. The recoveries for the volatile chloroform and carbon tetrachloride were about 90 percent, as expected. The less-volatile chlorobenzene and bromobenzene exhibited correspondingly poorer recoveries. These compounds are generally considered only marginally purgeable from water, so these results from milk are not surprising.

The methodology validation experiment indicated that the proposed method of analyzing human milk for volatile organic compounds was adequate. Sensitivity and detection limits were determined by the capabilities of the GC/MS/COMP system.

DEVELOPMENT OF ANALYTICAL PROTOCOL FOR SEMIVOLATILES

The extraction and cleanup method was validated using six model compounds (2,4-dichlorophenol, pentachlorobenzene, 1,2,3,4-tetrachloronaphthalene, 4,4'-dibromobiphenyl, 2,2',5,5'-tetrabromobiphenyl, and octachloronaphthalene) which were representative of the semivolatile (nonpurgeable) compounds of interest. The compounds were spiked into raw cow's milk at a level of about 1 μg/mL. Raw cow's milk was chosen as the closest readily available analog to mother's milk.

The results are presented in Table 13. The overall mean recovery was about 70 percent and the mean of the relative standard deviations was 22

Table 12. METHOD VALIDATION RECOVERY OF SELECTED VOLATILE STANDARDS FROM MILK

Compound[a]	b.p. (°C)	Percent Recovered[b]	Percent Retained[b]	Percent Accounted for[c]
^{14}C-chloroform	62	88 ± 5	6 ± 0.3	94 ± 2
^{14}C-carbon tetrachloride	76	88 ± 6	3 ± 3	91 ± 3
^{14}C-chlorobenzene	132	63 ± 2	26 ± 3	89 ± 1
^{14}C-bromobenzene	156	35 ± 3	51 ± 13	86 ± 10

[a] 80,000-94,000 dpm added to each sample.

[b] Mean ± standard deviation of three replicates.

[c] Sum of percent recovered and percent retained.

Table 13. METHOD VALIDATION RECOVERY OF SEMIVOLATILE COMPOUNDS SPIKED INTO RAW COW'S MILK

Compound	mp (°C)	bp (°C)	Concentration in Milk (ng/mL)	Mean Recovery (%)[a]	Standard Deviation (%)	Relative Standard Deviation[b] (%)
2,4-Dichlorophenol	45	207	1.12	59	12	20
Pentachlorobenzene	85	277	1.24	76	19	24
1,2,3,4-Tetrachloronaphthalene	197		1.37	59	15	25
4,4'-Dibromobiphenyl	164	357	1.04	58	19	33
2,2',5,5'-Tetrabromobiphenyl			0.93	94[c]	10	11
Octachloronaphthalene	198	441	1.08	78[c]	14	17

[a] Seven replicates.

[b] Standard deviation divided by mean multiplied by 100.

[c] Six replicates.

percent. These results indicated that refinements in the method should be considered prior to a large-scale study.

Two methods were available for removing fat and other nonvolatile components of the milk extract: Florisil column chromatography and gel permeation chromatography (GPC). Evaluation of the two techniques indicated that the Florisil method was more suitable to this project. The Florisil method was faster and had greater sample capacity than the GPC. In addition, the GPC procedure required the use of a pumping system, UV detector, and expensive, fragile GPC columns. Initial tests with both methods revealed interference problems, although those with GPC were more severe. Using GPC, decabromobiphenyl and hexabromobiphenyl eluted with the fat peak. This was judged totally unsatisfactory. Using Florisil, some fat eluted in the fraction with the compounds of interest, but repetition of the procedure yielded samples sufficiently clean for analysis.

DEPARTURES FROM THE ANALYTICAL PROTOCOLS

Emulsions

The formation of an emulsion during the toluene-acetone extraction of semivolatiles (step 6, Appendix C) was an area of concern. Approximately 80 percent of the time an emulsion occurred. To eliminate this, three approaches were taken with reasonable success. The first was to avoid the emulsion formation by swirling rather than shaking the toluene and acetone extracts. The second approach was to break the emulsion by adding Na_2SO_4 and waiting. Both the amounts of Na_2SO_4 and the time required varied. In severe cases emulsions were broken by filtering through glass wool wetted with toluene.

Lipid Removal Using Florisil

Problems were also encountered during the Florisil cleanup. Some samples had a tendency to solidify while concentrating the ether/pentane eluate, apparently due to abnormally high fat content. This usually occurred when the sample volume reached 1-3 mL. The samples to which this happened were diluted with pentane and eluted through another Florisil column. The Florisil cleanup was repeated until the samples remained liquid at small (<1.0 mL) volumes. Three cleanups was the maximum required for any sample.

GC/MS ANALYSIS PROCEDURES

Samples were analyzed by gas chromatography/mass spectrometry using an LKB 2091 EI/CI GC/MS. Operating conditions for the analysis of purgeables is given in Table 14 and the operating conditions for the extractables is given in Table 15. Analysis of the purgeables involved the use of the desorption apparatus described in Appendix B.

Quantitation of the unknowns was accomplished using relative molar responses (RMRs) as discussed in Appendices B and C. The RMRs were calculated from replicate determinations of known amounts of standards and analytes.

Qualitative Analysis

Initial identification of compounds by GC/MS involved comparisons of unknown spectra with data compiled in the Eight Peak Index of Mass Spectra(67). If the peaks present in the unknown spectra clearly matched the peaks of the standard compound in the tables and the intensities were about the same, then a positive identification was usually made. If peak intensities of unknowns varied from those of the standards, and there were isomers of the compounds that were not listed in the Eight Peak Index, then the compound was listed as an "isomer."

When the background peaks interfered with the spectrum of an unknown to an extent that made identification uncertain, the compound identification was labeled as "tentative" (tent.). If no standard spectra similar to those of the unknowns appeared in the mass spectral references, but fragments characteristic of a certain class of compounds were identified, tentative identifications were made on the basis of the characteristic fragments and apparent molecular weights. These identifications were also labeled "tent". Usually tentative identifications involved alkyl derivatives or homologs of classes of compounds that were positively identified in the same sample.

Positive identifications, as well as some tentative identifications, often required more detailed investigations of standard spectra in the Registry of Mass Spectral Data(68) or standard spectra found in other literature such as scientific journals. The Registry of Mass Spectral Data presents data in the form of histograms rather than as a list of peaks and their intensities. This type of format allowed more subtle differences in mass spectra to be considered when several similar standard spectra in the

Table 14. OPERATING CONDITIONS FOR GC/MS ANALYSIS OF PURGEABLES

Instrument	LKB 2091
Column	80m - SE-30 WCOT Capillary Column
Flow	1.7 mL/min He
Desorption Temperature	270°C
Desorption Time	8 min
Desorption Flow	15 mL/min He
Column Temperature	30°C for 2 min programmed to 240°C at 4°C/min
Scan Range	5 → 490 Dalton
Scan Speed	0 → 670 in 2 sec
Scan Cycle	1.7 sec
Injector Temperature	250°C
Accelerating Voltage	3500 V
Ionizing Energy	70 eV
Trap Current	50 μA
Source Temperature	210°C

Table 15. OPERATING CONDITIONS FOR THE GC/MS ANALYSIS OF SEMIVOLATILES

Instrument	LKB 2091
GC Column	25m SE-52 WCOT capillary column
Flow	1.5 mL/min with 15:1 split
Column Temperature	80°C for 3 min then 8°C/min to 265°C
Scan Range	5 → 530 Dalton
Scan Speed	2 sec 0 → 670 Dalton
Scan Cycle	2.4 sec
Injector Temperature	240°C
Accelerating Voltage	3500 V
Ionizing Energy	70 eV
Trap Current	50 μA
Source Temperature	210°C

Eight Peak Index appeared to represent possible candidates for unknown identifications.

A large number of sample components remained unidentified. These unidentified components were labeled "unknown."

In order to quantify the degree of certainty with which a compound has been identified, a "level" heirarchy has been established. The compound identification criteria are listed below:

Level I	Computer Interpretation. The raw data generated from the analysis of samples are subjected to computerized deconvolution/library search. Compounds identified using this approach have the lowest level of confidence. In general Level I is reserved for only those cases where compound verification is the primary intent of the qualitative analysis.
Level II	Manual Interpretation. The plotted mass spectra are manually interpreted and compared to those spectra compiled in a data compendium by a skilled interpreter. In general a minimum of five masses and intensities (±5 percent) should match between the unknown and the library spectrum. This level does not utilize any further information such as retention time since the authentic compound may not be available for establishing retention times.
Level III	Manual Interpretation Plus Retention Time/Boiling Point of Compound. In addition to the effort described under Level II, the retention time of the compound is compared to the retention time that has been derived from previous chromatographic analysis. Also the boiling point of the identified component is compared to the boiling points of other compounds in the near vicinity of the one in question when a capillary coated with a nonpolar phase has been used.
Level IV	Manual Interpretation Plus Retention Time of Authentic Compounds. Under this Level, the authentic compound has been chromatographed on the same capillary column using identical operating conditions and the mass spectrum of the authentic compound is compared to that of the unknown.
Level V	Level IV Plus Independent Confirmation Techniques. This Level utilizes other physical methods of analysis such as GC/Fourier transform infrared spectrometry, GC/high resolution mass spectrometry, or nmr analysis. This Level constitutes the highest degree of confidence in the identification of organic compounds.

Unless otherwise stated, all identifications in this report were Level II.

SECTION 7
RESULTS

VOLATILES

All 42 of the purged samples were analyzed by thermal desorption/GC/MS. The mass spectra from selected samples were interpreted manually to determine which compounds should be quantitated. From these data, selected compounds were quantitated in all samples. All data were stored on magnetic tape for subsequent processing and are routinely archived for at least 5 years.

Qualitative Identifications

Eight samples were interpreted. The results are presented in Appendix D. Samples were selected according to the following criteria. At least two samples were required from each collection site (Jersey City and Bayonne, NJ, were counted as two separate sites). The total ion current chromatograms were inspected and the samples with the greatest number of peaks or those containing very intense unique peaks (not observed in other samples) were selected. For those samples selected, all of the mass spectra were printed and interpreted manually by experienced spectroscopists.

Table 16 summarizes the compounds found and their frequency of occurrence. It is interesting to note that some compounds (e.g. 1,1,1-trichloroethane and hydrocarbons) are common air pollutants, others (e.g., dibromochloromethane) are common water pollutants, others (dimethyldisulfide, furans, aldehydes) appear to be metabolites, others (chlorofluorocarbons, siloxanes) are known background interferents, and others (iodopentane) are of unknown source.

Quantitation

Based upon the qualitative identifications summarized above, nine compounds were selected for quantitation in all of the samples. The results for four compounds are summarized in Table 17. As discussed below, the

Table 16. SUMMARY OF QUALITATIVE IDENTIFICATIONS OF VOLATILE COMPOUNDS IN MOTHER'S MILK[a]

	Sample Number[b]							
Compound	1081	1040	1107	1115	2048	2071	3053	3111
Halogenated Compounds								
chlorodifluoromethane	-	-	+	-	-	-	-	-
chlorotrifluoromethane	+	+	-	-	+	-	+	-
dichlorodifluoromethane	-	-	+	-	-	+	-	-
chloromethane	-	-	-	+	-	-	+	-
chloroethane	-	-	+	-	-	+	-	-
trichlorofluoromethane	+	+	+	+	+	+	-	+
dichloroethylene	-	+	-	-	-	-	-	-
Freon 113	+	+	+	+	+	+	+	+
methylene chloride	+	+	+	+	+	+	+	+
chloroform	+	+	+	+	+	+	-	+
1,1,1-trichloroethane	+	+	+	+	+	+	+	+
carbon tetrachloride	-	+	+	+	-	+	-	+
trichloroethylene	+	+	+	+	+	+	+	+
chloropentane	+	+	-	-	-	-	-	-
dibromochloromethane	-	-	-	-	-	+	-	-
tetrachloroethylene	+	+	+	+	+	+	-	+
dichloropropene	-	-	-	+	-	-	-	-
chlorobenzene	+	-	+	+	+	+	-	-
chlorohexane	+	+	+	-	+	-	-	-
iodopentane	-	-	-	+	-	-	-	-
3-methyl-1-iodobutane	+	+	-	-	-	-	-	-
chloroethylbenzene	-	-	-	+	-	-	-	-
dibromodichloromethane	-	-	-	+	-	-	-	-
dichlorobenzene	+	+	+	+	+	+	+	+
chlorodecane	+	-	-	-	-	-	-	-
trichlorobenzene	-	-	-	-	-	+	-	-
Aldehydes								
acetaldehyde	+	-	+	-	+	+	-	-
methylpropanal	-	+	+	-	-	-	-	-
n-butanal	+	-	+	+	-	+	+	+
methylbutanal	-	+	-	+	-	-	-	-
crotonaldehyde	-	-	-	+	-	-	-	-
n-pentanal	+	-	+	+	+	+	+	+
n-hexanal	+	+	+	+	+	+	+	+
furaldehyde	-	-	-	+	-	-	+	-
n-heptanal	+	+	+	+	+	+	+	-
benzaldehyde	+	+	+	+	+	+	+	+
n-octanal	+	-	+	+	-	-	+	-
phenyl acetaldehyde	-	-	-	+	-	-	-	-

(continued)

Table 16 (cont'd.)

Compound	Sample Number[b]							
	1081	1040	1107	1115	2048	2071	3053	3111
n-nonanal	+	+	+	+	+	-	+	-
methyl furaldehyde	-	-	-	-	-	-	+	-
n-decanal	-	-	-	+	-	-	+	-
n-undecanal	-	-	-	+	-	-	+	-
n-dodecanal	-	-	-	-	-	-	+	-
Ketones								
acetone	+	+	+	+	+	+	+	+
methyl ethyl ketone	+	+	-	-	+	+	+	-
methyl isopropyl ketone	-	-	-	+	-	+	-	-
methyl vinyl ketone	-	-	+	-	-	-	-	-
ethyl vinyl ketone	+	+	+	+	-	-	+	-
2-pentanone	+	+	+	+	-	-	-	-
methyl pentanone	-	-	+	+	-	-	-	-
methyl hydrofuranone	-	-	-	+	-	-	-	-
2-methyl-3-hexanone	-	-	-	+	-	-	-	-
4-heptanone	-	-	+	-	-	-	-	-
3-heptanone	+	-	+	-	+	+	-	-
2-heptanone	+	+	+	+	+	+	-	-
methyl heptanone	-	-	-	+	-	+	-	-
furyl methyl ketone	-	-	-	+	-	-	-	-
octanone	+	-	-	+	-	-	-	-
acetophenone	+	+	+	+	+	+	+	+
2-nonanone	+	-	+	+	-	+	-	-
2-decanone	-	-	-	+	-	-	-	-
alkylated lactone	-	-	-	+	-	-	-	-
phthalide	-	-	+	-	-	-	-	-
Other Oxygenated Isomers								
C_4H_6O	-	-	-	-	-	-	+	-
C_4H_8O	-	-	-	-	-	+	+	-
$C_5H_{10}O$	-	-	+	-	+	+	+	+
C_6H_8O	-	-	-	-	-	-	+	-
$C_6H_{10}O$	-	-	+	-	-	-	+	-
$C_4H_6O_2$	+	-	-	-	-	-	-	-
$C_6H_{12}O$	+	-	-	-	+	-	-	-
$C_7H_{12}O$	-	-	+	+	-	-	+	+
$C_7H_{10}O$	-	-	+	+	-	-	-	-
$C_7H_{14}O$	-	-	-	-	+	-	+	-
$C_6H_6O_2$	-	-	-	-	-	-	+	-
$C_8H_{14}O_2$	-	-	+	-	-	-	-	-
$C_8H_{16}O$	+	-	-	-	+	-	-	-
$C_7H_8O_2$	-	-	-	+	-	-	+	-
$C_7H_{10}O_2$	-	-	-	-	-	-	+	-

(continued)

Table 16 (cont'd.)

Compound	Sample Number[b]							
	1081	1040	1107	1115	2048	2071	3053	3111
Other Oxygenated Isomers (continued)								
$C_9H_{18}O$	+	-	-	-	+	-	+	-
$C_8H_6O_2$	-	-	-	-	-	-	+	-
$C_{10}H_{12}O$	-	-	+	-	-	-	-	-
$C_{10}H_{14}O$	-	-	-	-	+	-	-	-
$C_{10}H_{16}O$	-	-	-	+	-	-	+	-
$C_{10}H_{18}O$	-	-	+	+	-	+	-	-
$C_{10}H_{20}O$	+	-	-	-	-	-	+	-
$C_{10}H_{22}O$	-	-	-	-	+	-	-	-
$C_9H_8O_2$	-	-	-	-	-	-	+	-
$C_{11}H_{20}O$	-	-	-	-	-	-	+	-
$C_{10}H_{10}O_2$	-	-	-	-	-	-	+	-
Alcohols								
methanol	-	-	-	-	-	+	-	-
isopropanol	+	+	+	+	+	+	+	+
2-methyl-2-propanol	-	-	-	-	-	+	-	-
_n_-propanol	-	-	-	-	-	+	-	-
1-butanol	-	-	+	+	+	-	-	-
1-pentanol	-	+	-	+	+	+	-	-
α-furfuryl alcohol	-	-	-	+	-	-	+	-
2-ethyl-1-hexanol	-	-	-	-	+	-	-	-
phenol	-	-	+	-	-	+	-	-
2,2,4-trimethylpentyl-1,3-diol	-	-	-	+	-	-	-	-
α-terpineol	-	-	-	-	+	-	-	-
Acids								
acetic acid	-	-	-	+	-	-	+	-
decanoic acid	-	-	-	-	+	-	-	-
Sulfur Compounds								
sulfur dioxide	-	-	-	-	-	-	-	+
carbon disulfide	+	+	+	+	+	+	+	+
dimethyl disulfide	-	+	+	+	-	+	+	+
carbonyl sulfide	-	-	-	+	-	-	-	-

(continued)

Table 16 (cont'd.)

Compound	Sample Number[b]							
	1081	1040	1107	1115	2048	2071	3053	3111
Nitrogen Compounds								
nitromethane	-	-	+	-	-	-	-	-
$C_5H_6N_2$	-	-	-	+	-	-	-	-
$C_5H_8N_2$	-	-	-	+	-	-	-	-
$C_4H_4N_2O$	-	-	-	+	-	-	-	-
methyl acetamide	-	-	+	-	-	-	-	-
benzonitrile	-	-	+	+	-	+	-	-
methyl cinnoline	-	-	-	+	-	-	-	-
Esters								
vinyl propionate	-	+	+	+	-	-	-	-
ethyl acetate	-	-	-	-	-	+	-	-
ethyl-*n*-caproate	-	-	-	-	-	+	-	-
methyl caprylate	-	-	-	-	-	+	-	-
ethyl caprylate	-	-	-	-	-	+	-	-
isoamyl formate	-	+	-	-	-	-	-	-
methyl decanoate	-	-	-	-	-	+	-	-
ethyl decanoate	-	-	-	-	-	+	-	-
Ethers								
dimethyl ether	-	+	-	-	-	-	-	-
p-dioxane	-	-	+	-	-	-	-	-
dihydropyran	-	-	+	+	-	-	-	-
Epoxide								
1,8-cineole	-	-	-	+	-	-	-	-
Furans								
furan	-	-	-	-	-	-	+	-
tetrahydrofuran	-	-	+	-	-	-	-	-
methyl furan	-	-	-	+	-	-	+	-
methyl tetrahydrofuran	-	+	-	-	-	-	-	-
ethylfuran	-	-	+	+	-	-	-	-
dimethylfuran	-	-	-	+	-	-	-	-
2-vinylfuran	-	-	-	-	-	-	+	-
furaldehyde	-	-	-	+	-	-	+	-
2-*n*-butylfuran	-	+	-	-	-	-	-	-
2-pentylfuran	+	+	+	+	+	+	+	-
methylfuraldehyde	-	-	-	-	-	-	+	-
furyl methyl ketone	-	-	-	+	-	-	-	-
α-furfuryl alcohol	-	-	-	+	-	-	+	-
benzofuran	-	-	+	+	-	+	-	-

(continued)

Table 16 (cont'd.)

Compound	Sample Number[b]							
	1081	1040	1107	1115	2048	2071	3053	3111
Alkanes								
C_3H_8	-	-	+	-	-	-	-	-
C_4H_{10}	+	+	+	-	+	+	+	-
C_5H_{12}	+	+	+	+	+	+	+	+
C_6H_{14}	+	+	+	+	+	+	+	+
C_7H_{16}	+	+	+	+	-	+	+	+
C_8H_{18}	+	+	+	+	+	-	+	+
C_9H_{20}	+	+	+	+	+	+	+	+
$C_{10}H_{22}$	+	-	+	+	+	+	+	+
$C_{11}H_{24}$	+	-	+	+	+	+	+	+
$C_{12}H_{26}$	+	-	+	+	+	+	+	+
$C_{13}H_{28}$	-	+	-	-	+	-	+	-
$C_{14}H_{30}$	-	-	-	+	+	-	+	-
$C_{15}H_{32}$	-	-	-	+	-	-	+	-
Alkenes								
C_3H_6	+	-	-	-	-	+	-	-
C_4H_8	+	-	+	-	+	+	-	+
C_5H_{10}	-	-	+	-	+	-	-	+
C_6H_{12}	+	+	+	+	+	+	+	+
C_7H_{14}	+	+	+	+	+	+	+	+
C_8H_{16}	+	+	+	+	+	+	+	+
C_9H_{18}	+	+	+	+	-	+	+	+
$C_{10}H_{20}$	-	+	+	+	+	+	+	-
$C_{11}H_{22}$	+	+	+	+	-		+	-
$C_{12}H_{24}$	-	-	+	-	-	-	-	-
$C_{13}H_{26}$	-	-	-	-	-	-	+	-
isoprene	-	+	-	-	-	-	-	-
Alkynes								
C_5H_8	-	-	-	-	-	+	-	+
C_6H_{10}	-	-	-	-	+	-	-	-
C_7H_{12}	+	-	-	-	+	-	+	-

(continued)

Table 16 (cont'd.)

Compound	Sample Number[b]							
	1081	1040	1107	1115	2048	2071	3053	3111
Alkynes (continued)								
C_8H_{14}	-	+	-	+	-	-	+	-
C_9H_{16}	+	-	-	+	+	-	+	-
$C_{10}H_{18}$	-	-	+	+	-	-	-	-
$C_{12}H_{22}$	-	-	-	+	-	-	-	-
Cyclic Hydrocarbons								
cyclopentane	+	+	+	+	-	+	-	+
methylcyclopentane	+	-	+	-	+	+	+	+
cyclohexane	+	+	+	-	+	+	-	-
ethylmethylcyclohexane	-	-	+	-	-	-	-	-
$C_{10}H_{14}$ isomers	+	-	-	-	-	-	-	-
$C_{10}H_{16}$ isomers (other)	+	+	-	-	+	+	-	-
limonene	+	+	+	+	+	+	+	+
methyldecalin	-	-	+	-	-	-	-	-
α-pinene	-	-	+	-	-	-	-	-
camphene	-	-	-	-	-	+	-	-
camphor	-	-	-	-	-	+	-	-
Aromatics								
benzene	+	+	+	+	+	+	+	+
toluene	+	+	+	+	+	+	+	+
ethylbenzene	+	+	+	+	+	+	+	+
xylene	+	+	+	+	+	+	+	+
phenylacetylene	-	-	-	+	-	-	-	-
styrene	+	+	+	+	+	+	+	+
benzaldehyde	+	+	+	+	+	+	+	+
C_3-alkylbenzene isomers	+	+	+	+	+	+	+	+
C_4-alkylbenzene isomers	-	+	+	+	+	+	+	-
methylstyrene	-	-	+	-	-	+	-	-
dimethylstyrene	+	+	-	+	+	+	-	-
C_5-alkylbenzene isomers	-	-	+	+	-	-	-	-
naphthalene	+	+	-	+	+	+	+	-
C_6-alkylbenzene isomers	-	-	-	+	-	-	-	-

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[a] Arranged by class in approximate elution order. See Appendix D for sample-by-sample identifications. + = present; - = not identified in sample.

[b] Participant code number.

Table 17. VOLATILES QUANTITATED IN MOTHER'S MILK SAMPLES (ng/mL)

Site	Sample Number[a]	Chloroform[b]	Tetrachloro-ethylene	Chlorobenzene	Dichloro-benzene[c]
Bayonne, NJ	1016	-[d]	1.5	0.2	6.7
	1032	0.3	1.5	0.1	9.1
	1040	0.1	1.1	0.1	66
	1057	0.7	0.9	0.1	0.2
	1073	0.7	3.8	0.1	2.2
	1081	1.3	6.3	0.1	32
Jersey City, NJ	1024	13	43	0.1	2.8
	1107	17	7.4	0.2	68
	1115	1.7	8.1	0.3	49
	1123	20	17	0.1	2.2
	1164	65	4.0	0.1	0.9
Pittsburgh, PA	2014	0.9	0.8	0.2	0.2
	2022	1.5	1.8	0.1	1.1
	2048	0.6	1.8	0.1	8.9
	2055	0.8	1.0	0.05	0.7
	2063	0.6	1.6	0.1	3.1
	2071	1.2	1.0	0.1	1.4
	2089	0.7	26	0.2	0.5
	2097	6.7	1.8	-	0.3
	2105	2.8	1.3	0.4	1.1
	2113	1.2	0.7	0.1	0.4
	2121	0.8	2.4	TR[e]	2.0
	2139	0.6	0.7	0.1	0.9
Baton Rouge, LA	3012	2.9	0.1	0.3	4.2
	3020	0.7	0.5	0.1	0.6
	3038	0.8	1.7	0.2	1.3
	3046	21	2.5	0.1	2.2

(continued)

Table 17 (cont'd)

Site	Sample Number[a]	Chloroform[b]	Tetrachloro-ethylene	Chlorobenzene	Dichloro-benzene[c]
	3053	0.3	0.4	0.2	1.8
	3079	0.8	0.6	0.1	0.2
	3087	0.7	0.4	0.2	5.2
	3095	1.3	1.0	0.3	4.2
	3103	0.6	0.2	0.1	>22
	3111	1.8	0.5	-	44
Charleston, WV[g]	4010	5.0	1.2	0.1	0.7
	4028	7.2	1.4	0.2	1.9
	4036	7.5	3.9	10	0.2
	4051	8.2	0.6	0.2	1.1
	4069	-	0.4	0.1	3.6
	4085	5.3	0.4	-	3.8
	4093	12	1.0	0.1	0.04
	4101	8.7	1.0	0.1	26
	4119	11	>19[f]	0.04	1.4

[a] Participant code number.

[b] See text for caveats with respect to chloroform.

[c] All isomers summed.

[d] Not detected.

[e] Trace.

[f] Instrument saturated.

[g] Sample 4044 lost due to instrumental malfunction.

quantitation of the other five compounds is not reported, since the levels in milk were not judged sufficiently greater than background to be reliable.

Upon inspection, it is obvious that most values are low relative to only a few high "outliers." These high values suggest that there is a range of levels of these compounds which may correlate with exposure. These results were analyzed statistically to determine if any of the values correlated significantly. As can be seen in Table 18, the arithmetic mean and median values generally are quite different. The arithmetic mean is skewed toward the high end, generally due to one or two relatively high values. A more realistic representation of the central data is the geometric mean. These geometric mean values were tested for their significance (i.e., are the geometric means significantly different from site to site?). Table 19 summarizes this data. From this table, it appears that samples from Jersey City have significantly higher levels of chloroform, tetrachloroethylene, and dichlorobenzene than the other study samples. Charleston samples appear to have significantly higher levels of chloroform, and Bayonne samples appear to have significantly higher levels of dichlorobenzene.

To test if any of the compound levels were related, the Spearman correlation coefficients (nonparametric correlation based on the sample, designed to lessen the weight of a single high outlier) were determined as shown in Table 20. There does not appear to be any compound-to-compound correlation among the subjects.

In interpreting these data, it must be remembered that this is a very small data set. Therefore these data should not be used to extrapolate to the city or area from which the samples were collected.

Quality Control

Table 21 presents the quality control results for chloroform, tetrachloroethylene, chlorobenzene, and dichlorobenzene. The very high recovery of chloroform from the controls indicates either a miscalculation of the amount actually spiked or contamination of the samples used as controls. Since the procedural blanks contained about 15 times less chloroform, the former explanation is most reasonable. However, the chloroform values reported in Table 17 must be interpreted subject to the following

Table 18. SUMMARY STATISTICS FOR VOLATILE COMPOUNDS BY SITE[a]

Site	Chloroform	Tetrachloroethylene	Chlorobenzene	Dichlorobenzene
Bayonne, NJ				
Maximum	1.3	6.3	0.2	66
Mean[b]	0.52	2.52	0.12	19.37
Median	0.5	1.5	0.004	7.9
S.D.	0.48	2.13	0.1	25.54
n	6	6	6	6
Jersey City, NJ				
Maximum	65	43	0.3	68
Mean[b]	23.34	15.9	0.16	24.48
Median	17	8.1	0.1	2.8
S.D.	24.3	15.9	0.089	31.69
n	5	5	5	5
Pittsburgh, PA				
Maximum	6.7	26	0.4	8.9
Mean[b]	1.53	3.41	0.12	1.71
Median	0.85	1.45	0.1	1
S.D.	1.74	7.13	0.11	2.41
n	12	12	12	12
Baton Rouge, LA				
Maximum	21	2.5	0.3	44
Mean[b]	3.09	0.79	0.16	8
Median	0.8	0.5	0.15	3.2
S.D.	6.34	0.75	0.096	13.98
n	10	10	10	10
Charleston, WV				
Maximum	12	>19	10	26
Mean[b]	7.21	3.21	1.20	4.30
Median	7.5	1	0.1	1.4
S.D.	3.55	6.02	3.30	8.25
n	9	9	9	9
Overall				
Maximum	65	43	10	68
Mean[b]	5.57	4.10	0.37	9.15
Median	1.25	1.25	0.1	1.95
S.D.	10.9	8.15	1.53	17.3
n	42	42	42	42

[a] Maximum, mean and median values are ng/mL.

[b] Arithmetic mean.

Table 19. SIGNIFICANCE OF THE DIFFERENCES IN THE GEOMETRIC MEANS BY SITE

Site	Geometric Mean (ng/mL)			
	Chloroform	Tetrachloroethylene	Chlorobenzene	Dichlorobenzene
Bayonne	0.45	2.09	0.12	8.33
Jersey City	14.7	11.5	0.16	8.55
Pittsburgh	1.23	1.82	0.12	1.21
Baton Rouge	1.53	0.67	0.15	3.83
Charleston	5.92	1.65	0.42	1.98
Significance[a]	0.01	0.01	N.S.[b]	0.05

[a] 0.01 implies 99 percent confidence that the numbers are statistically different, while 0.05 implies 95 percent confidence.

[b] Not significant.

Table 20. SPEARMAN CORRELATION COEFFICIENTS FOR VOLATILE ORGANICS FOUND IN MOTHER'S MILK

	Chloroform	Tetrachloro-ethylene	Chlorobenzene	Dichloro-benzene
Chloroform	1.0	0.37[a]	-0.02[b]	-0.13[b]
Tetrachloro-ethylene		1.0	0.007[b]	0.05[b]
Chlorobenzene			1.0	0.03[b]
Dichloro-benzene				1.0

[a] Significant at 0.05 level (95 percent confidence).

[b] Not significant
Sample size = 42

Table 21. QUALITY CONTROL RESULTS FOR VOLATILES IN MILK

Type of Sample	Chloroform	Tetrachloroethylene	Chlorobenzene	Dichlorobenzene
Blanks[a]				
n	7	7	7	17
Mean (ng/mL)[b]	1.2	0.22	0.03	0.12
S.D.	1.3	0.11	0.025	0.19
RSD (%)	108	49	84	159
Controls[c]				
n	8	8	8	0[d]
Mean Recovery[e]	14.02[f]	1.12	0.62	-
S.D.	8.20	0.41	0.34	-
RSD (%)	58	37	55	-

[a] Blanks consisted of two field water blanks and five water blanks purged with the milk samples to monitor procedural background. No difference between the two types of blanks was observed.

[b] Arithmetic mean.

[c] Controls consisted of two spiked raw cow's milk samples carried to the field and returned, two spiked raw cow's milk samples stored in the laboratory, two spiked water samples carried to the field and returned, and two spiked water samples stored in the laboratory. No major differences were observed between the four types of samples. Samples were spiked at 30-90 ng/volume purged (or about 1 ng/mL).

[d] Not included in control spiking solution.

[e] 1.0 = 100 percent recovery.

[f] Extremely high recovery probably a result of improper loading of controls.

considerations: the mean reported levels in the samples were only 4.9 times the blank levels; the recovery from controls was about 1400 percent, invalidating the recovery study; and chloroform is known to be a laboratory atmospheric contaminant.

The compounds presented in Table 17 represented significant levels above the background in blanks. Several other compounds were quantitated that did not exhibit substantial concentrations. These compounds, with the ratio of the mean in the samples to the mean in the background given in parenthesis, were: 1,1,1-trichloroethane (1:1), benzene (2:1), toluene (2:4), trichloroethylene (1:2) and carbon tetrachloride (1:4). These levels in the samples cannot be reliably assigned to either the milk sample or to laboratory contamination. If these compounds are present in milk, they are very low and cannot be regarded as significant, given the limitations of the technique employed. Apparently, mother's milk does not represent a bioconcentration matrix for these compounds.

SEMIVOLATILES

Three samples were fully interpreted, as presented in Appendix E. As can be seen from the data, few compounds of interest were observed in the mass spectra. The data were searched on the GC/MS data system for target compounds (PCNs, PBBs and PCBs) using single ion plots called up from the full data set. No evidence for any of these compounds was observed at a detection limit of about 20 ppb. DDE was quantitated in five samples as shown in Table 22. These values were in the range generally reported by previous investigators (see Tables 2 - 4). Since none of the target compounds were present in detectable quantities, no further identification or quantitation was attempted.

Table 22. DDE AND TETRACHLOROBIPHENYL LEVELS IN SELECTED MOTHER'S MILK SAMPLES

Site	Sample Number	ng/mL Milk	
		DDE	Tetrachlorobiphenyl
Pittsburgh	2105	45	ND[b]
Pittsburgh	2121	73	T[c]
Charleston, WV	4069	107	ND
Charleston, WV	4085	38	ND
Charleston, WV	4093	91	ND
	Mean[d]	71	-
	S.D.	29	
	RSD (%)	42	
	Median	73	

[a] Samples selected as having the most intense total ion current chromatograms.

[b] Not detected.

[c] Trace.

[d] Arithmetic mean.

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APPENDIX A

DATA COLLECTION INSTRUMENTS

STUDY OF ORGANIC COMPOUNDS IN HUMAN MILK

EPA Contract No. 68-01-3849
RTI Project No. 31U-1521-22

DATA COLLECTION INSTRUCTIONS

Performed for

Office of Toxic Substances
Environmental Protection Agency
Washington, DC 20460

1.0 Introduction

Under contract to the Office of Toxic Substances, Environmental Protection Agency (EPA), the Research Triangle Institute (RTI) is conducting a limited study designed to measure environmental pollutant levels in human milk and to evaluate the utility of using this body fluid in specific pollutant studies for populations in the vicinity of manufacturing plants and/or industrial user facilities. RTI is responsible for all phases of the study, including study design, subject recruitment, chemical analysis of milk samples, and report writing. RTI is a not-for-profit contract research organization located in North Carolina's Research Triangle Park between Raleigh, Durham, and Chapel Hill. The Institute was incorporated as a separate operating entity in 1958 by the University of North Carolina (UNC) at Chapel Hill, Duke University at Durham, and North Carolina State University at Raleigh, and is still closely affiliated with the three universities.

2.0 Overview

Four urban areas have been chosen as performance sites; they are Bridgeville, Pennsylvania; the area which includes Linden and Bayonne, New Jersey and western Staten Island, New York; Baton Rouge, Louisiana; and South Charleston and Nitro, West Virginia. These sites represent high-probability areas for the presence of one or more of the chemicals of interest in human milk. The selected industrial chemicals of interest include polychlorinated naphthalenes, tetrachlorethylene, trichloroethane, dichloropropane, benzene, polybrominated biphenyls, chlorinated phenols, toluene, chlorinated benzenes, and chloroform.

At each of the four sites, arrangements will be made to work through clinical facilities such as hospitals, clinics, or physician's offices, in order to recruit a panel of respondents. At each site ten participants will be recruited, for a total of 40. Potential participants (lactating females) will be screened to determine that they live in one of the areas of interest and are willing and able to provide the milk sample.

A questionnaire will be administered for each participant to obtain information on demographic variables, residence histories, and potential exposure situations; for each participant, a sample of milk will be collected and analyzed for the compounds of interest by gas chromatography/mass spectrometry or high pressure liquid chromatography. A professional member of the facility's staff, such as a registered nurse, will be trained in the proper procedures to administer the questionnaire and obtain the milk sample. To try to reduce the non-participation rate due to refusals, and to reimburse the subject for the time spent on the study, volunteers will be offered a $5.00 incentive for participating.

3.0 Data Collection

3.1 General Remarks

Data collection for this research effort consists of the following steps:

1. Screening of potential participants (lactating women) to determine that they live in one of the areas of interest (see below), that they have resided in that area for at least the preceding 12 months, that they have remained in that area continuously for the preceding week, and that they are willing and able to provide a milk sample.

2. When an eligible person is encountered, the nature and purpose of the study will be explained and their participation solicited.
3. When an eligible person agrees to participate, the person will be required to sign a Participant Consent Form (PCF) in order to participate in the study.
4. Once the participant has signed the PCF, the person should be listed on the Participant Listing Form (PLF), a Patient Number assigned, and the data collector will proceed to administer the Study Questionnaire (SQ) and collect the milk sample.
5. Once the SQ has been administered and the milk sample collected, the participant will be offered a $5.00 incentive for participating.
6. Milk samples and completed data collection instruments will be returned to RTI.

3.2 Survey Instruments

As indicated in the preceding section, there are 3 data collection instruments for this research effort, the PCF, the PLF, and the SQ; subsequent sections contain instructions for the use of each instrument as well as item-by-tiem explanations for their completion, and general descriptions are provided below. The survey instruments have been designed hopefully to provide an efficient means of collecting and recording the requisite data for the study. *It is imperative that all survey instruments be completed accurately.* The success and reliability of the study and its results are dependent upon the quality of data collected, which will be fully dependent on the accuracy of your execution of your assignment. As you complete a form, conduct a thorough edit to verify that required data have been entered and entered correctly. Copies of the data collection instruments appear in Attachment 1.

3.2.1 Participant Consent Form (PCF)

- Purpose: The purposes of the PCF are to introduce the study; explain its objectives, sponsorship (the relationship and roles of RTI and EPA), and requirements of and risks, burdens, and benefits to participants; and stress that participation is completely voluntary and that all data collected will be kept confidential.
- General Description: The PCF is a single page form printed on special paper which makes three copies from a single impression. The survey title appears at the top, along with the name of RTI; spaces for necessary identifying information appear at the bottom.
- Administration: The PCF will be signed by the participant and contains an agreement to provide the necessary information and milk sample. Participants may freely withdraw from the study at any time; however, in order to encourage participation RTI offers an incentive of five dollars to each participant to be paid after each data set (PCF, SQ, and milk sample) is obtained. Again, confidentiality of data is stressed, including steps

taken to disassociate the name of the participant from the data once collected; for example, the PCF is the *only* data collection instrument which bears the name of the participant and allows its association to study identification numbers, but will be maintained in hard copy only and stored in a restricted area. To further emphasize this disassociation, the incentive will be paid in cash rather than by check or money order, although the participant will sign the PCF indicating that the incentive was received. *A signed PCF must be obtained for each participant before proceeding with Study Questionnaire (SQ) administration and collection of the milk sample.*

- Disposition: The top (white) copy will be attached to the appropriate SQ until it is received at RTI and verified; the yellow copy will be provided to the participant; the pink copy will be retained by the data collector.

3.2.2 Participant Listing Form (PLF)

- Purpose: The purpose of the PLF is to provide a means of assigning unique numbers to participants at each performance site.
- General Description: The PLF is a single page form printed on pink paper; space for Comments is provided on the reverse side. The survey title appears at the top, along with the names and addresses of RTI and EPA/OTS and a confidentiality statement.

- Administration: As each participant is enlisted up to the required number (10), that participant should be listed on the PLF.
- Disposition: When data collection at a site or facility is completed, the PLF (or a copy) should be sent to RTI.

3.2.3 Study Questionnaire (SQ)

- Purpose: The purpose of the SQ is to obtain information on participants, including demographic characteristics such as age, sex, race, and occupation; residence information; health information such as current health status and prescription medications; and personal characteristics such as hobbies.
- General Description: The SQ is divided into six sections, dealing respectively with demographic characteristics, occupation, health and personal habits, residence and household information, information on the interviewer and respondent, and information regarding the milk sample, including an indication as to whether or not the milk sample was obtained, the date and time of acquisition of the sample, and the date the sample was shipped to RTI. Participants will be identified by a unique study number used to correlate and cross-identify the questionnaires and samples by way of pre-printed self-adhesive labels. The SQ is 5 pages long, with space provided for comments.

- **Administration**: An SQ is to be completed for each participant for whom a signed PCF is obtained.
- **Disposition**: The SQ's are to be sent to RTI as instructed.

3.3 Screening

As indicated in section 3.1, potential participants (lactating women) should be screened to determine that they meet certain study criteria for participation:

1. That they are willing and able to provide a milk sample of sufficient quantity (approximately 100 ml.),
2. That they live in one of the areas of interest (see below),
3. That they have resided in that area for at least the preceding 12 months, and
4. That they have remained in that area continuously for the preceding 7 days.

As indicated in section 2.0, four areas have been chosen as performance sites, with a specific *Site Number* assigned to each which will remain constant for each site and is to be entered where appropriate on data collection instruments as follows:

Site	Site Number
Northern New Jersey/Staten Island, New York	1
Bridgeville, Pennsylvania	2
Baton Rouge, Louisiana	3
Nitro/South Charleston, West Virginia	4

With the exception of Bridgeville, Pennsylvania, participants residing in some areas at each site are of considerably more interest to the study than those living in others, as discussed in the following sections.

3.3.1 Northern New Jersey/Staten Island, New York

Within the Northern New Jersey/Staten Island area, potential participants residing in some communities are of more interest than those residing in others, more or less in the order listed below:

1. Bayonne, NJ
2. Northern Staten Island (Port Richmond), NY
3. Linden, NJ
4. Carlstadt, NJ
5. Saddle Brook, NJ
6. Jersey City, NJ
7. Kearney, NJ
8. Newark, NJ
9. Elizabeth, NJ
10. Sayreville, NJ
11. Rahway, NJ
12. Edison, NJ
13. Parlin, NJ
14. Passaic, NJ
15. Patterson, NJ
16. Wayne, NJ

3.3.2 Baton Rouge, Louisiana

Potential participants residing in Baton Rouge are of primary interest to this study; other communities in the Baton Rouge area of interest are Placquemine, St. Gabriel, and Geismar.

3.3.3 Nitro/South Charleston, West Virginia

Potential participants residing in Nitro and South Charleston are of primary interest to this study; other communities of interest in the area are Belle and Institute.

3.4 Participant Listing Form

When an eligible person is encountered who agrees to participate, that person should be listed on a PLF in order to be assigned a unique *Participant Number*. The PLF is completed by entering the appropriate *Site Number* (see section 3.3 above); then, each time that an eligible participant is encountered who agrees to participate, up to the number required, enter the *Participant's Name (Last, First, Middle)* on the PLF and assign a *Participant Number* in the left-hand column, beginning with 0001 at each site unless otherwise instructed. Assign Participant Numbers consecutively for all study participants. Where appropriate, enter the participant's *Medical Record Number* in the right-hand column. *When making numerical entries, right-adjust and enter leading zeros.*

3.5 Participant Consent Form

Potential participants must understand exactly what is involved in participation in the study and what benefits may be realized by participation; this understanding and agreement must be documented by a signed PCF. In the event that the potential participant is under the age of 18 years, the person's parent or other legal guardian must sign the PCF in order for the designated eligible to participate.

More specifically, the potential participant and/or that person's parent, guardian or other spokesman, must understand that full participation in the study consists of providing answers to a questionnaire related to environmental exposure, part of which relates to the individual's household in general and part of which is related to the individual participant *(be prepared to show the person the SQ)*, and providing a milk sample of approximately 100 ml. *(be prepared to show the person one of the collection bottles.)* The individual must further understand that she will only enjoy certain limited benefits in return for her time and inconvenience, primarily a $5.00 incentive to be disbursed after administration of the questionnaire and collection of the milk sample. The individual must understand that participation in the study is completely *voluntary* and that she may withdraw at any time, but that *payment of the incentive is dependent on full participation.* The individual must also understand that all data collected in the study will be held strictly confidential, and that names will not be disclosed.

If the participant or that perons's parent, guardian or other spokesman agrees to participate, read through the PCF with them and make entries where appropriate. At the bottom, record the *Date (month, day, and year)* that the PCF is signed and print the *Participant's full Name (First, Middle or Maiden, Last - do not abbreviate)*; record the appropriate *Site Number* (see section 3.3 above) and *Participant Number* (from the PLF); have the participant (or other appropriate person) sign the PCF; enter your signature as witness; and record the participant's home *Address (Street Number and Name, City, State, and Zip Code)* in the spaces provided.

After data collection (administration of SQ and collection of milk sample) is completed, the participant (or that person's parent or guardian) should be given $5.00. The recipient must sign in the space provided at the bottom of the PCF to indicate receipt of the incentive. Should the signatures on the PCF for *Participant* and *Recipient* be other than the participant's, please explain in the Comments section of the SQ.

Finally, as indicated in section 3.2.1, the top (white) copy of the PCF is to be attached to the appropriate SQ; the yellow copy is to be provided to the participant or her guardian; and the pink copy is to be retained by the data collector.

3.6 Study Questionnaire

Before proceeding with administration of the SQ, *read the justification and confidentiality statement* in the box on the cover. Enter the appropriate *Site* (see section 3.3 above) *and Participant* (from the PLF) *Numbers*. Stapled inside the SQ you will find a set of pre-printed, self-adhesive labels which are necessary to identify corresponding SQs and samples. Each label contains a unique *Study Number*, which should be the same on all labels in a set, and an indication of what the label is for. You should also have some labels that have only a Study Number and a few that are completely blank; these are for your use in the event that a label is damaged or missing. If you use a label that has a Study Number only, you will have to write on the label what it is intended for, such as *MILK;* if you use a blank label, you must write on the label the Study Number and what it is intended for. Check to be sure that all the labels in a given SQ contain the same Study Number; if not, do not use the SQ and return it to RTI. If the Study Number is the same on all labels, remove the one for the *QUESTIONNAIRE* and place it on the cover of the SQ over the spaces provided for the *Study Number*. Space for *Comments* is provided on page 5.

If the participant is under 18 years of age, the SQ may have to be administered in whole or part to the parent or guardian, and *must be administered in that person's presence*. If the participant suffers from a speech or hearing deficit, or is otherwise incapacitated, the SQ may have to be administered to the spouse or some other spokesman.

Item 1 - Race: Indicate the participant's *race* by placing an X in the appropriate box. This question may be answered by observation; however, if there is *any doubt whatsoever*, ask.

Item 2 - Age: Determine and enter the participant's *age* in years as of the last birthday.

Item 3 - Birthdate: Determine and enter the participant's exact *birthdate* (month, day and year). Again, remember to right-adjust and enter leading zeros. *A note on dates: accept and record partial dates, if that is all that the respondent can provide;* in that case, indicate missing elements of the date with a dash (-) — for example, April 1977 would be recorded as $\boxed{0}\boxed{4}$ - $\boxed{-}\boxed{-}$ - $\boxed{7}\boxed{7}$.

Item 4 - Weight: Determine and enter the participant's approximate weight in *pounds (to the nearest pound--no fractions!) or kilograms,* in which case observe the decimal.

Item 5 - Height: Determine and enter the participant's approximate height in *inches or centimeters.*

Item 6 - Current Employment: Determine if the participant is currently employed in any capacity and place an X in the appropriate box. If the answer is Yes, continue to Item 7; if the answer is No, skip to Item 10.

Item 7 - Length of Present Employment: Determine and record the length of time that the participant has been employed by her present employer; enter the units in the spaces provided and then place an X in the appropriate box to indicate whether the units represent days, months, or years.

Item 8 - Occupation Away From Home: Determine if the participant's occupation *usually* takes her away from home and place an X in the appropriate box. If Yes, continue to Item 9; if No, skip to Item 11. This question, and Item 9 below, are aimed at eliciting information regarding the location of the participant's various exposure to the environment.

Item 9 - Location of Present Employment: If the participant is currently employed, determine the nature (not the name) and location (street address, city, state, and Zip Code, if known) of the employer. By *nature,* we mean the type of business, such as service station, school, hospital, grocery store, doctor's office, hotel, restaurant, etc.

Item 10 - Employment Status: If the participant is not presently employed, determine which of the provided categories best describes the participant's status and place an X in the appropriate box. If the response is choice 1 or 2, skip to Item 15; if the response is choice 3-5, continue to Item 11.

Item 11 - Usual Occupation: Determine and record the participant's *usual (or most common)* occupation *(when employed);* be succinct - e.g., high school coach, waitress, hotel desk clerk, taxi driver.

Item 12 - Present Occupation: Determine if the participant is presently employed in her usual occupation (indicated in Item 11) and place an X in the appropriate box. Items 12 and 13 may be skipped for unemployed, retired and disabled persons.

Item 13: If the response to Item 12 was positive, determine how long the participant *has been* employed in her usual occupation (recorded in Item 11) and record; enter the units in the spaces provided and then place an X in the appropriate box to indicate whether the units represent days, months or years.

Item 14: Determine if the participant presently works at or in any of the listed occupations or establishments and place an X in each appropriate box.

Item 15 - Present Smoking Status: Ascertain if the participant currently smokes *cigarettes,* and place an X in the appropriate box. If YES, continue to Item 16; if NO, skip to Item 18.

Item 16 - Age at First Smoke: If the participant is a smoker (a positive response to Item 15), ascertain the age (in years) at which the participant started smoking and record in the spaces provided.

Item 17 - Smoking Frequency: Ascertain how many cigarettes the participant smokes *per day, on the average,* and place an X in the appropriate box. *If the participant uses tobacco in some form other than cigarettes, such as snuff, record in the space provided.*

Item 18 - Time Outdoors: Ascertain the *average number of hours* that the participant spends *out of doors each day* and record in the spaces provided — another indication of environmental exposure.

Item 19 - Time Away From Home: Determine how many hours of the day *on the average* the participant normally spends more than 2 miles away from home, and record in the spaces provided. This determination should be done separately for *weekdays* and *weekends*.

Item 20 - General Health Status: Using the four qualifiers provided, ascertain the participant's general current health status and place an X in the appropriate box.

Item 21 - Prescription Medications: Inquire as to whether the participant is currently taking any *prescription medication(s)* on a regular daily basis and place an X in the appropriate box; if YES, determine and record the *drug name* - e.g., penicillin, oral contraceptives, Valium, phenobarbital, etc.

Item 22 - Non-prescription Medications: Inquire as to whether the participant has taken any *non-prescription medications* in the past 24 hours, and place an X in the appropriate box; If YES, determine and record the *drug name* -e.g., aspirin, vitamins, Dristan, Bufferin, Alka-Seltzer, etc.

Item 23 - Gasoline: Inquire as to whether the participant pumps her own gasoline, for example at *self-service* pumps, and place and X in the appropriate box.

Item 24 - Egg Consumption: Determine and record the approximate number of *eggs* that the participant has eaten in the past 48 hours. Again, in recording numerical entries, remember to right-adjust and enter leading zeros.

Item 25 - Hobbies: Determine if the participant pursues any of the listed avocations and place an X in each appropriate box.

Item 26: Determine if the participant pursues *any* activity that includes *regular* use of solvent glue or model airplane cement, and place an X in the appropriate box.

Item 27 - Length of Residence in Area: Determine how many years the participant has lived in the *area of interest*, and record in the spaces provided. Round to the nearest year, except that if the response is less than one year record as $\boxed{<}\boxed{1}$ and terminate the interview; the individual is ineligible to participate further in the study. This situation should be detected during the screening process.

Item 28 - Length of Residence at Current Address: Determine how long the participant has lived at her *current address;* record the units in the spaces provided and place an X in the appropriate box to indicated whether the units represent days, months, or years. Use the most appropriate units and round to the nearest appropriate unit. For example, more than 28 days should be expressed in months and more than 11 months should be expressed in years. If the participant has resided at her current address for less than 12 months, but has lived in the area of interest for at least 12 months, record any previous addresses during the preceding 12 months (city and state is sufficient) in the Comments section.

Item 29 - Cooling Appliances: Determine whether any of the indicated appliances or others, in which case *specify,* are used to cool the participant's home and place an X in the appropriate box(es) for *all that apply.*

Item 30 - Home Garden: Determine if the participant's household consumes food grown in a home garden and indicate the response by placing an X in the appropriate box. If a positive response is obtained, determine the *location* of the garden and record. Location could be *participant's backyard,* or another community, in which case specify city and state; be as specific as possible.

Item 31 - Commercial Food Source: Determine where the participant's household *usually* obtains fruit and/or vegetables and record.

Again, be as specific as possible. For example, if the city or town has more than one store by the same name, the store name alone would not be an adeuqate answer; as a matter of course, record the name and location of the store, market, or vendor.

Items 32-34 - Water Sources: In Item 32, try to determine the *primary* source of *drinking water* for the participant's household and place an X in the appropriate box. In Item 33, determine if the *same* primary drinking water source indicated in Item 32 is used for *drink mixes* such as coffee and tea; if it differs, indicate how. In Item 24, try to determine the primary source of *water for cooking* in the participant's household and place an X in the appropriate box. For example, some households in some areas of the country use *bottled water* for drinking and drink mixes but *tap water* (from whatever source) in cooking.

Item 35 - Other Household Tobacco Use: Inquire as to whether *other members* of the participant's household smoke, and place an X in *the* appropriate box; if YES, determine if the other members smoke cigarettes, cigars, a pipe, etc. and place an X in *each* appropriate box.

Item 36 - Occupation of Other Household Members: Determine if any other members of the participant's household work at any of the listed occupations or businesses, and place an X in each appropriate box.

Item 37 - Hobbies of Other Household Members: Determine if any other members of the participant's household pursue any of the listed avocations, and place an X in each appropriate box.

Respondent/Interviewer Information

Item 38 - Respondent: Indicate, by placing an X in the appropriate box, whether the person who served as the *primary respondent* was the participant or some other person, in which case *specify* in the space provided.

Item 39 - Interviewer Number: Enter your assigned 3-digit *Interviewer* identification *Number*.

Item 40 - Date of Interview: Enter the date (month, day and year) that the interview was conducted and the questionnaire completed.

Item 41 - Interviewer Name: The name of the person administering the questionnaire should be printed in the space provided.

Sample Information

Item 42: Indicate, by placing an X in the appropriate box, whether or not a milk sample was collected; if not, explain in the Comments section below.

Item 43 - Date and Time of Milk Sample Collection: If a milk sample is collected, record the *date (month, day and year)* and approximate *time (using a 24-hour clock)* of such collection. The time should correspond to the time that collection was completed; on a 24-hour clock, add 12 to the p.m. hours - e.g., 1:00 p.m. would be 13:00, 5:30 would be 17:30, etc.

Item 44 - Date Shipped to RTI: Record the date (month, day and year) that the respective milk sample was shipped to RTI, or turned over to an RTI representative.

3.7 Collection of the Milk Sample

3.7.1 General Remarks

As indicated in section 1.0 above, the milk samples are being collected for chemical analysis by RTI as part of an EPA study to measure pollutant levels in human milk and evaluate the utility of using this body fluid in specific pollutant studies. The chemical compounds for which the samples will be analyzed are present in extremely low levels, so the utmost care and cleanliness must be used to prevent either contamination or loss. The instructions below are designed to preserve the integrity of the sample and should be followed precisely.

3.7.2 Sample Collection Instructions

1. The bottles provided have been thoroughly cleaned and should be kept tightly closed, except during sampling; do not wash or otherwise clean them.
2. Remove the *MILK SAMPLE* label from the sheet of labels in the appropriate SQ and place on one of the collection bottles.
3. The milk should be *manually* expressed directly into the the bottle; do not use breast pumps or other devices as the plastics in such devices would contaminate the sample. Hands should be cleaned and thoroughly rinsed to remove any residual soap; do not use rubber gloves.

4. Collect as much milk as possible. Unless the mother has recently nursed her infant, at least half a bottle should be easily obtainable. Less than half a bottle is unuseable and does not constitute a sample. The ability of the participant to provide an adequate sample should be determined during the screening process.
5. Immediately cap the bottle and double check to see that the study numbers on the bottle and questionnaire match.
6. The milk sample should be immediately frozen following collection and remain so until shipping.
7. Note any deviations from this procedure in the Comments section of the appropriate SQ.

3.7.3 Shipping Instructions

1. Pack the container as it was received.
2. Fill the can with *dry ice*.
3. Make sure that there is adequate padding to prevent breakage, that all excess space is filled with packing material.
4. Fill out enclosed Federal Express forms, attach to the outside of the box, and seal the box.
5. Call Federal Express and have them pick up the package.
6. When Federal Express picks up the package, call Dr. Mitch Erickson at RTI (see below) to notify him that Federal Express has picked up the package; if Dr. Erickson is out, leave an appropriate message with his secretary.

7. Mail the corresponding questionnaires to RTI in one of the envelopes provided.
8. When the questionnaires are in the mail, call Ben Harris at RTI (see below) to notify him that the questionnaires are in the mail; if Mr. Harris is out, leave an appropriate message with his secretary.

4.0 Confidentiality

All survey research conducted by RTI is based on highest ethical standards, including those related to confidentiality. These standards are applied from the earliest steps of deciding whether or not RTI should participate in a proposed survey to the final steps of analyzing and reporting the information obtained. Strict precautions must be observed at all times to protect the rights of those whom we interview or about whom we collect data. Such precautions are built into the study design, so that promises of confidentiality and anonymity will be upheld during all phases of data handling and analysis.

No amount of effort to insure confidentiality will be successful, however, unless those responsible for data collection in the field maintain equally rigid standards, treating with utmost confidence all information offered *or observed* during data collection. Successful and meaningful survey research is dependent on the establishment of trust between individuals engaged in data collection and sources of information, and maintaining this sense of responsibility to the public throughout all survey activities.

Each data collector will be required to sign in duplicate a contractual agreement which includes provisions on confidential treatment of data. This agreement is designed to protect you as well as RTI and participating institutions and individuals. A copy of this agreement appears in Attachment 2.

The importance of total confidentiality cannot be over-emphasized. Any breach of confidence could result in litigation.

5.0 Contacts with Project Staff

During the data collection period it will be necessary for data collectors to maintain regular contact with RTI project staff by telephone. While you are collecting data, problems or confusing issues may arise that are not addressed in these instructions. You are encouraged to telephone RTI whenever you experience a problem or encounter a situation which you feel you cannot adequately handle.

All supplies required for data collection will be furnished by RTI. Should you require additional supplies during the conduct of data collection, inform your RTI contact so that proper arrangements can be made. Need for additional supplies should be anticipated so that your work will not be delayed while you await receipt of needed items. All study-related items that are in your possession at the conclusion of data collection are to be returned to RTI or disposed of according to instructions from your RTI contact.

Calls to RTI should be made between the hours of 8:30 a.m. and 5:00 p.m. (Eastern Time), Monday through Friday, to RTI's toll-free number, 800-334-8571. Request to speak to the appropriate project staff member listed below:

Dr. Mitch Erickson *(regarding milk sample collection)*	Extension 6505
Mr. Ben Harris *(regarding participant selection and questionnaire administration)*	Extension 6055

If the problem is particularly acute, and you have trouble getting through on the toll-free line, call *collect* 919-541-6505 (Dr. Erickson) or 919-541-6055 (Mr. Harris). After 6:00 p.m. Eastern Time you may call Mr. Harris *collect* at work (919-541-6055) or person-to-person at home (919-942-6988).

Attachment 1

Data Collection Instruments

OMB No. 158-37801
Approval Expires September 198

RESEARCH TRIANGLE INSTITUTE
STUDY OF ORGANIC COMPOUNDS IN HUMAN MILK

PARTICIPANT CONSENT FORM

I understand that Research Triangle Institute is engaged in a study of various organic compounds as they appear in human milk. I understand that the survey is being conducted in order to measure the levels of various organic compounds in human milk, and is limited to the purpose stated. I further understand that the survey is being conducted under the auspices of the United States Environmental Protection Agency in cooperation with

[Name of Local Agency] .

I do hereby freely consent to participate in this study of organic compounds in human milk and understand that my participation will consist of providing answers to a questionnaire related to environmental exposure and providing a milk sample of approximately 100 ml. I understand that an agent of Research Triangle Institute will administer the questionnaire and collect the milk sample, after which I will receive an incentive of five dollars for my participation.

I understand that my name will not be voluntarily disclosed, or referred to in any way when compiling and evaluating the results of the study. I understand that participation in this study may result in no direct benefits to me, other than those described herein, and that I am free to withdraw from this study at any time. It has been explained to me that there are no significant risks to me from participation in this study. I further understand that while participating in the study I will be free to ask any questions concerning the study; if I have any further questions about the project, I know that I am free to contact

______________________________ telephone number ______________

or Mr. Benjamin S. H. Harris, III, Survey Operations Center, Research Triangle Institute, Research Triangle Park, North Carolina 27709, telephone number 919-541-6055.

Date: ☐☐ - ☐☐ - ☐☐
(Month) (Day) (Year)

Participant's Name: ______________________________
(Print)

Site Number: ☐

Participant Number: ☐☐

SIGNATURES:

Participant: ______________________ Witness: ______________________

Address: ____
(Street Number and Name)

____ ☐☐☐☐☐
(City) (State) (Zip Code)

Incentive Received: ______________________________
(Signature of Recipient)

STUDY OF ORGANIC COMPOUNDS IN HUMAN MILK

Sponsored by:
Office of Toxic Substances
Environmental Protection Agency
Washington, D.C. 20460

Conducted by:
Research Triangle Institute
P.O. Box 12194
Research Triangle Park, North Carolina 27709

PARTICIPANT LISTING FORM

NOTICE: All information recorded on this document which would permit identification of an individual or an establishment will be held in strict confidence, will be used only by persons engaged in and for the purposes stated for this study, and will not be disclosed or released to other persons or used for any other purpose.

Site Number: ☐

Participant Number				Participant Name *(Last, First, Middle)*	Medical Record Number							

COMMENTS

OMB No. 158-5780
Approval Expires September 10

STUDY OF ORGANIC COMPOUNDS IN HUMAN MILK

Sponsored by:
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Conducted by:
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QUESTIONNAIRE

THE RESEARCH TRIANGLE INSTITUTE OF RESEARCH TRIANGLE PARK, NORTH CAROLINA, IS UNDERTAKING A RESEARCH STUDY FOR THE U.S. ENVIRONMENTAL PROTECTION AGENCY OF LEVELS OF VARIOUS ORGANIC COMPOUNDS IN HUMAN MILK. THE INFORMATION RECORDED IN THIS QUESTIONNAIRE WILL BE HELD IN STRICT CONFIDENCE AND WILL BE USED SOLELY FOR RESEARCH INTO THE EFFECTS OF ENVIRONMENTAL FACTORS ON PUBLIC HEALTH. ALL RESULTS WILL BE SUMMARIZED FOR GROUPS OF PEOPLE; NO INFORMATION ABOUT INDIVIDUAL PERSONS WILL BE RELEASED WITHOUT THE CONSENT OF THE INDIVIDUAL. THIS QUESTIONNAIRE IS AUTHORIZED BY LAW (P.L. 94-469). WHILE YOU ARE NOT REQUIRED TO RESPOND, YOUR COOPERATION IS NEEDED TO MAKE THE RESULTS OF THIS SURVEY COMPREHENSIVE, ACCURATE, AND TIMELY.

Study number: ☐☐☐☐☐☐ Site number: ☐ Participant number: ☐☐

First, I would like to ask some general questions about you.

1. Race:
☐ 1 Hispanic
☐ 2 American Indian/ Alaskan Native
☐ 3 Black, not of Hispanic origin
☐ 4 Asian/Pacific Islander
☐ 5 White, not of Hispanic origin
☐ 6 Other *(Specify)* ____________

2. What was your age in years at last birthday? ☐☐ Years

3. What is your birthdate?
(Month) ☐☐ - *(Day)* ☐☐ - *(Year)* ☐☐

4. What is your approximate weight?
☐☐☐ lbs. ☐☐☐.☐ kgs.

5. What is your height? ☐☐ inches ☐☐☐ cms.

Next, I would like to ask some questions about your occupation.

6. Are you presently employed in any capacity? ☐ 1 Yes *(Continue)* ☐ 2 No *(Go to Q. 10)*

7. How long have you been employed by your present employer? ☐☐ Units ☐ 1 Days ☐ 2 Months ☐ 3 Years

8. Does your occupation usually take you away from home? ☐ 1 Yes *(Continue)* ☐ 2 No *(Go to Q. 11)*

9. What is the nature and location (street address) of the company for which you work?
(Specify) ____ ☐☐☐☐☐ *(Zip Code)*

10. If not presently employed, which of the following best describes your status?
☐ 1 Housewife
☐ 2 Student
} *(Go to Q. 15)*
☐ 3 Unemployed
☐ 4 Retired
☐ 5 Disabled
} *(Continue)*

11. What is/was your usual occupation? *(Specify)* ____

12. Are you presently employed in this occupation? ☐ 1 Yes ☐ 2 No

13. If yes to above question, how long have you been employed in that occupation?
(Questions 12 and 13 may be skipped for unemployed, retired, and disabled persons.) ☐☐ Units ☐ 1 Days ☐ 2 Months ☐ 3 Years

14. Do you work at or in any of the following occupations or establishments? *(Check all that apply.)*
☐ 1 Painting
☐ 2 Dry cleaning
☐ 3 Chemical plant
☐ 4 Petroleum plant
☐ 5 Service station/garage/engine repair
☐ 6 Furniture refinishing or repair

Next, I would like to ask some questions regarding your health and personal habits.

15. Do you smoke? ☐1 Yes *(Continue)* ☐2 No *(Go to Q. 18)*

16. How old were you when you first started smoking? ☐☐ Years

17. On the average, how many cigarettes do you smoke per day?

☐1 Less than ¼ pack (1-4 cigarettes)
☐2 About ½ pack (5-14 cigarettes)
☐3 About 1 pack (15-24 cigarettes)
☐4 About 1½ packs (25-34 cigarettes)
☐5 About 2 packs (35-49 cigarettes)
☐6 More than 2 packs (50 or more cigarettes)

NOTE: *If the participant uses tobacco in some other form (other than cigarettes—e.g., snuff), record here:* ______________________________

18. What is the average number of hours than you spend out of doors each day? ☐☐ Hours

19. How many hours of the day, on the average, do you normally spend away from home? *(Average separately for weekdays and weekends).*

Weekdays ☐☐ Hours Weekends ☐☐ Hours

20. What do you consider the current status of your health? *(Check one.)*

☐1 Excellent ☐2 Good ☐3 Fair ☐4 Poor

21. Are you currently taking any prescription medication(s) on a regular daily basis? ☐1 Yes ☐2 No

If yes, specify: ______________________________

22. Have you taken any non-prescription medications in the past 48 hours? ☐1 Yes ☐2 No

If yes, specify: ______________________________

23. Do you pump your own gas? ☐1 Yes ☐2 No

24. How many eggs have you eaten in the past 48 hours? ☐☐

25. Do you pursue any of the following hobbies? *(Check all that apply.)*

☐1 Furniture refinishing ☐2 Painting ☐3 Scale models ☐4 Gardening

26. Do you pursue any activity that includes regular use of solvent glue or model airplane cement?

☐1 Yes ☐2 No

Lastly, I would like to ask some questions about your residence and household.

27. How many years have you lived in this area? ☐☐ Years

28. How long have you lived at your current address? ☐☐ Units ☐ 1 Days ☐ 2 Months ☐ 3 Years

29. Do you cool your home with any of the following appliances? *(Check all that apply.)*

- ☐ 1 Central air conditioning
- ☐ 2 Window air conditioner(s)
- ☐ 3 Evaporative cooler(s)
- ☐ 4 Window fan(s)
- ☐ 5 Ceiling exhaust fan(s)
- ☐ 6 Circulating fan(s)
- ☐ 7 None of these
- ☐ 8 Do not know
- ☐ 9 Other *(Specify)* ____________________

30. Does your household grow any of its own food in a home garden? ☐ 1 Yes ☐ 2 No ☐ 3 Do not know

If yes, specify location of garden ____________________

31. Where does your household obtain fresh fruit and/or vegetables? *(Specify)* ____________________

32. What is the primary source of your water for drinking?

- ☐ 1 Bottled water
- ☐ 2 Tap - municipal supply
- ☐ 3 Tap - community well
- ☐ 4 Tap - private well
- ☐ 5 Tap - cistern
- ☐ 6 Do not know
- ☐ 7 Other *(Specify)* ____________________

33. Is that the same primary source of water for drink mixes such as coffee, tea, Kool-Aid, etc?

☐ 1 Yes ☐ 2 No If no, how does it differ? *(Specify)* ____________________

34. What is the primary source of your water for cooking?

- ☐ 1 Bottled water
- ☐ 2 Tap - municipal supply
- ☐ 3 Tap - community well
- ☐ 4 Tap - private well
- ☐ 5 Tap - cistern
- ☐ 6 Do not know
- ☐ 7 Other *(Specify)* ____________________

35. Does anyone else in your household smoke? ☐ 1 Yes ☐ 2 No ☐ 3 Do not know

If yes, check all that apply: ☐ 1 Cigarettes ☐ 2 Cigars ☐ 3 Pipe ☐ 4 Other *(Specify)* ____________________

36. Does anyone else in your household work at any of the following occupations/businesses? *(Check all that apply.)*

- ☐ 1 Painting
- ☐ 2 Dry cleaning
- ☐ 3 Chemical plant
- ☐ 4 Petroleum plant
- ☐ 5 Service station/garage/engine repair
- ☐ 6 Furniture refinishing or repair

37. Does anyone else in your household pursue any of the following hobbies? *(Check all that apply.)*

☐ 1 Painting ☐ 2 Furniture refinishing ☐ 3 Scale models ☐ 4 Gardening

---

RESPONDENT/INTERVIEWER INFORMATION

38. Respondent: ☐ 1 Participant ☐ 2 Other *(Specify)* ____________________

39. Interviewer number: ☐☐☐

40. Date of interview: *(Month)* ☐☐ - *(Day)* ☐☐ - *(Year)* ☐☐

41. Interviewer name: ____________________

SAMPLE INFORMATION

42. Was a milk sample collected? ☐ 1 Yes ☐ 2 No

43. If yes, date (Month) ☐☐ - (Day) ☐☐ - (Year) ☐☐ and time Hours : Minutes ☐☐ : ☐☐

44. Date shipped to RTI: (Month) ☐☐ - (Day) ☐☐ - (Year) ☐☐

COMMENTS

Attachment 2

Research Triangle Institute
Data Collection Agreement

Research Triangle Institute **DATA COLLECTION** **AGREEMENT**	For Project ______________ ______________ Project No. ______________

I, ______________________________, agree to provide, as an employee of Powerforce Company, Inc., field data collection services for Research Triangle Institute in connection with the project named above.

a. I agree to provide services within the guidelines and specifications for project data collection activities provided by Research Triangle Institute;

b. I am aware that the research being conducted by the Institute is being performed under contractual arrangement with ______________ ______________________________;

c. I agree to treat as confidential all information secured during interviews or obtained in any project-related way during the period I am providing services to the Institute;

d. I shall at all times recognize and protect the confidentiality of all information secured while providing my services throughout the conduct of this research project;

e. I am aware that the survey instruments completed form the basis from which all the analysis will be drawn, and therefore agree that all work for which I submit invoices will be of high quality and in accordance with project specifications; and

f. I fully agree to conduct myself at all times in a manner that will obtain the respect and confidence of all individuals from whom data will be collected and I will not betray this confidence by divulging information obtained to anyone other than authorized representatives of Research Triangle Institute.

Dated at ______________________________
(City/Town) (State)

this ______________ day of ______________ 19____

____
Employee

____
For Research Triangle Institute

Disposition: Original to RTI; yellow copy retained by Employee.

APPENDIX B

SAMPLING AND ANALYSIS OF VOLATILE ORGANICS IN MILK

SAMPLING AND ANALYSIS OF VOLATILE ORGANICS IN MILK

1.0 Principle of the Method

Volatile compounds are recovered from an aqueous or solid sample by warming the sample and purging helium over it. The vapors are then trapped on a Tenax cartridge which can be introduced by thermal desorption directly into the GC/MS for analysis. This protocol is the result of extensive development efforts.(1-9)

2.0 Range and Sensitivity

For a typical organic compound approximately 30 ng is required to obtain mass spectral identification using high resolution gas capillary GC/MS analysis. Based on a 50 g milk sample, a detection limit of about 0.6 μg/kg would be possible. The dynamic range (limit of detection to saturation on the mass spectrometer) for a purged sample is $\sim 10^4$; however, smaller samples may be purged and the upper end of the range increased commensurately.

3.0 Interferences

Two possible types of interferences must be considered: (1) material present in the sample which physically prevents the effective purge of the sample, and (2) material which interferes with the analysis of the purged sample. In the former case, several techniques have been developed to handle such problems (e.g., foaming) by diluting and stirring the sample. The second case is minimized by the use of GC/MS for the analysis, since unique combinations of m/z and retention time can be selected for most compounds. This permits the evaluation of compounds even though chromatographic resolution is not obtained.

4.0 Precision and Accuracy

The purge and trap technique has been evaluated for a variety of matrices using model compounds which are expected to be typical of volatile halogenated compounds.(1)

The recovery of the purge step was validated using cow's milk samples spiked with ^{14}C-chloroform, ^{14}C-carbon tetrachloride, ^{14}C-chlorobenzene and ^{14}C-bromobenzene. The average recoveries were 88, 88, 63, and 35 percent, respectively. The recoveries correlate roughly with volatility (inversely with boiling point), so anticipated recovery for other compounds may be interpolated from these data.

5.0 Apparatus

5.1 Purge Apparatus

The purge apparatus is shown in Figure 1.

5.2 Sampling Cartridges

The sampling tubes are prepared by packing a 10-cm long x 1.5-cm i.d. glass tube containing 6 cm of 35/60 mesh Tenax GC with glass wool in the ends to provide support.(2,3) Virgin Tenax is extracted in a Soxhlet extractor for a minimum of 24 h with redistilled methanol and pentane prior to preparation of cartridge samples.(2,3) After purification of the Tenax GC sorbent and drying in a vacuum oven at 100°C for 2-3 h all of the sorbent material is meshed to provide a 35/60 mesh-size range. Sample cartridges are then prepared and conditioned at 270°C with helium flow at 30 mL/min for 30 minutes. The conditioned cartridges are transferred to Kimax® (2.5 cm x 150 cm) culture tubes, immediately sealed using Teflon-lined caps, and cooled. This procedure is performed in order to avoid recontamination of the sorbent bed.(2,3)

5.3 GC/MS/COMP

The volatile halogenated hydrocarbons purged from water are analyzed on either an LKB 2091 GC/MS with an LKB 2031 data system or a Varian MAT CH-7 GC/MS with a Varian 620/i data system. The sample, concentrated on a Tenax GC cartridge, is thermally desorbed using an inlet manifold system.(2,4) The operating conditions for the thermal desorption unit and the analysis Tenax GC cartridges are given in Table 1.

6.0 Materials

6.1 Sampling

Clean, 120 mL, wide-mouth glass bottles with Teflon-lined caps are used for the collection of milk samples.

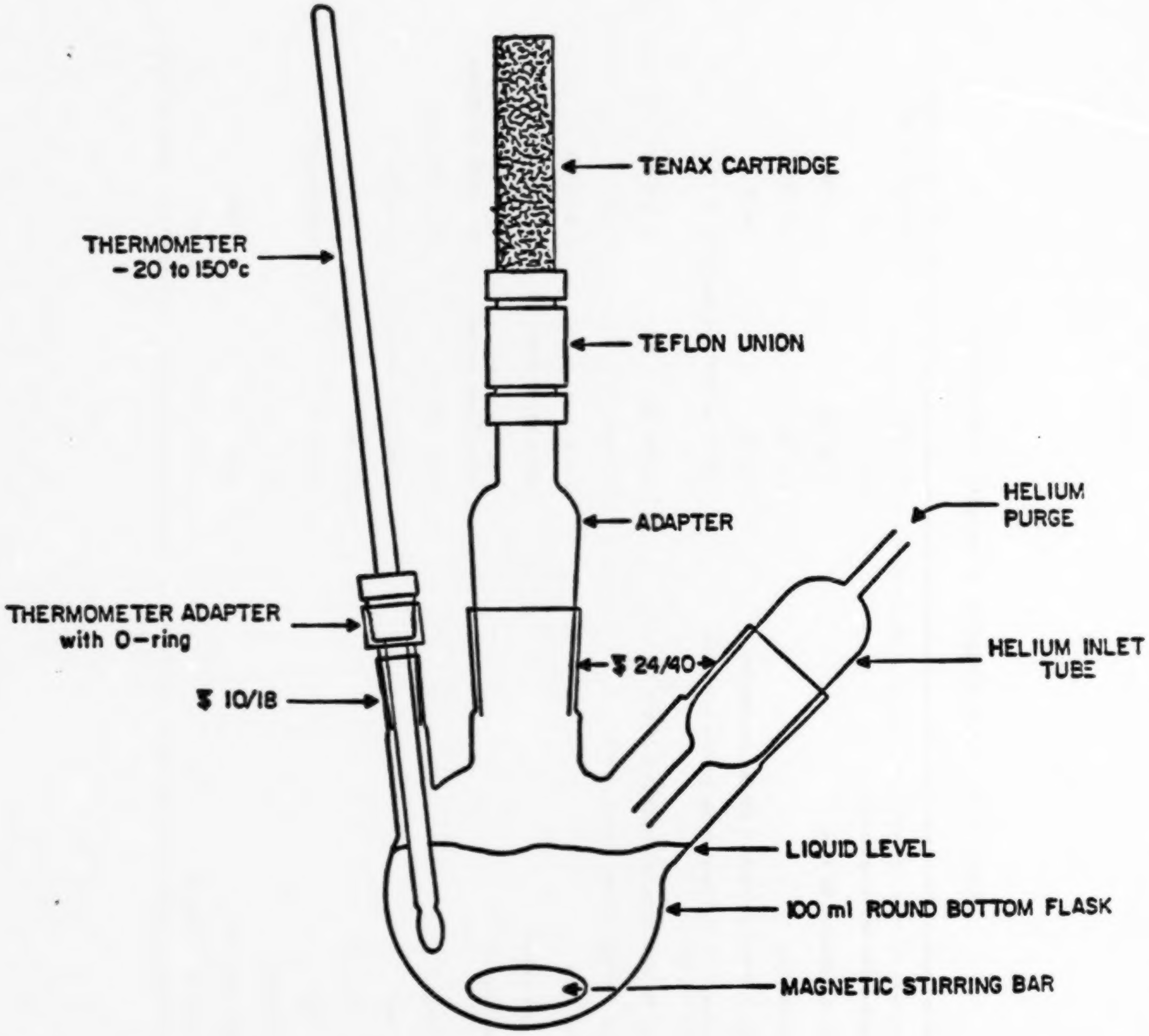
TENAX CARTRIDGE
THERMOMETER
- 20 to 150°c
TEFLON UNION
ADAPTER
HELIUM
PURGE
THERMOMETER ADAPTER
with O-ring
$ 24/40
HELIUM INLET
TUBE
$ 10/18
LIQUID LEVEL
100 ml ROUND BOTTOM FLASK
MAGNETIC STIRRING BAR

Figure B-1. Diagram of headspace purge and trap system.

Table B-1. INSTRUMENTAL OPERATING CONDITIONS

	LKB 2091	Varian MAT CH-7
Desorption chamber temperature	270	265
Desorption chamber He flow	15 mL/min	10 mL/min
Desorption time	8.0 min	8.0 min
Capillary trap temperature during desorption	-196°C	-196°C
Temperature of capillary trap during injection onto column	-196°C to 250°C - then held at 190°C	
Time of He flow through capillary trap	12 3/4 min	12 3/4 min
He flow through column [sweep time]	9.5 min	4 min
Carrier flow	2.0 mL/min	1.0 mL/min
Capillary column	100 m SE-30 SCOT	20 m SE-30 WCOT
Column temperature	30°C for 2 min, then 4°/min to 240°	20 → 240° at 4°/min
Scan range	5-490 dalton	20 → 500 dalton
Scan rate	2 sec full scale	1 sec/decade
Scan cycle time	2.4 sec	4.5 sec
Scan mode	parabolic	exponential
Trap current	4A	
Filament current	50μA	300μA
Accelerating volatage	3.5 kV	2kV

6.2 Purge

Tenax cartridges - 16-mm o.d. x 10.5 cm glass tubes filled with 6 cm of Tenax with 1-cm glass-wool plugs in each end.

Charcoal cartridges - 16-mm o.d. x 6 cm filled with 4 cm of charcoal and glass-wool plugs in each end.

Glass culture tubes with Teflon-lined screw caps.

7.0 Procedure

7.1 Collection of Field Samples

Milk (60-120 mL) is expressed directly into the wide-mouth bottle, capped tightly, and frozen for shipment and storage. To preserve the integrity with respect to volatiles, handling and transfer must be minimized.

7.2 Purging of Volatiles

The apparatus is assembled as depicted in Figure 1, including the Tenax GC cartridges (1.5-cm diameter x 6.0-cm length). A carbon cartridge 1.5-cm diameter x 4.0-cm length is connected to the effluent end of the Tenax cartridge to prevent contamination of the cartridge by laboratory vapors. The milk sample is cooled to ~4°C, shaken vigorously and 100 mL diluted with 350 mL distilled water. The pH of the solution is adjusted to 4.0 with sulfuric acid. A glass-wool plug is inserted into the center neck of the flask just above the level of the solution and, with the flask in a heating mantle, the solution is heated to 70°C while it is stirred with a magnetic stirrer. The sample is purged at 15 mL helium/min and 70°C for 90 minutes. The loaded cartridge is removed and stored in a culture tube containing 1-2 g $CaSO_4$ desiccant for 2-12 h. The desiccant is removed from the culture tube and the dry, loaded cartridge stored at -20°C.

7.3 Analysis of Sample Purged on Cartridge

The instrumental conditions for the analysis of volatile compounds of the sorbent Tenax GC sampling cartridge are shown in Table 1.(2-9) The thermal desorption chamber and six-port valve are maintained at 270°C and 200°C, respectively. The helium purge gas through the desorption chamber is adjusted to 15-20 mL/min. The nickel capillary trap at the inlet manifold is cooled with liquid nitrogen. In a typical thermal desorption cycle a sampling cartridge is placed in the preheated desorption chamber and helium gas is channeled through the cartridge to purge the vapors into the liquid nitrogen cooled nickel capillary trap. After desorption the six-port valve is rotated and the temperature on the capillary loop is rapidly raised; the carrier gas then introduces the vapors onto the high resolution GC column. The glass capillary column is temperature programmed from 20°C to 240°C at 4°/min and held at the upper limit for a minimum of 10 minutes. After all of the components have eluted from the capillary column, the analytical column is cooled to ambient temperature and the next sample is processed.

7.4 Quantitation

All data are acquired in the full scan mode. Quantitation of the halogenated compounds of interest is accomplished by utilizing selected ion plots (SIPs), which are plots of the intensity of specific ions (obtained from full scan data) versus time. Using SIPs of ions characteristic of a given compound in conjunction with retention times permits quantitation of components of overlapping peaks. Two external standards, perfluorobenzene and perfluorotoluene, were added to each Tenax GC cartridge in known quantities just prior to analysis. In order to eliminate the need to construct complete calibration curves for each compound quantitated, the method of relative molar response (RMR) is used. In this method the relationship of the RMR of the unknown to the RMR of the standard is determined as follows:

$$\mathrm{RMR}_{\mathrm{std}} = \frac{A_{\mathrm{unk}}/\mathrm{moles}_{\mathrm{unk}}}{A_{\mathrm{std}}/\mathrm{moles}_{\mathrm{std}}}$$

$$\mathrm{RMR}_{\mathrm{unk/std}} = \frac{A_{\mathrm{unk}}/g_{\mathrm{unk}}/\mathrm{GMW}_{\mathrm{unk}}}{A_{\mathrm{std}}/g_{\mathrm{std}}/\mathrm{GMW}_{\mathrm{std}}}$$

where A = peak response of a selected ion,
std = standard
unk = unknown
g = number of grams present, and
GMW = gram molecular weight.

Thus, in the sample analyzed:

$$g_{\mathrm{unk}} = \frac{(A_{\mathrm{unk}})(\mathrm{GMW}_{\mathrm{unk}})(g_{\mathrm{std}})}{(A_{\mathrm{std}})(\mathrm{GMW}_{\mathrm{std}})(\mathrm{RMR}_{\mathrm{unk/std}})}$$

The value of an RMR is determined from at least three independent analyses of standards of accurately known concentration prepared using a gas permeation system.(3) The precision of this method has been determined to be generally ±10 percent when replicate sampling cartridges are examined.

8.0 References

1. Michael, L. C., M. D. Erickson, S. P. Parks, and E. D. Pellizzari, Anal. Chem., 52, 1836-1841 (1980).
2. Pellizzari, E. D., "Development of Analytical Techniques for Measuring Ambient Atmospheric Carcinogenic Vapors," Publication No. EPA-600/2-76-076, Contract No. 68-02-1228, 185 (November 1975).
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4. Pellizzari, E. D., J. E. Bunch, R. E. Berkley and J. McRae, Anal. Chem., 48, 803 (1976).
5. Pellizzari, E. D., J. E. Bunch, B. H. Carpenter and E. Sawicki, Environ. Sci. Tech., 9, 552 (1975).
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8. Pellizzari, E. D., J. E. Bunch, R. E. Berkley and J. McRae, Anal. Lett., 9, 45 (1976).
9. Pellizzari, E. D., Analysis of Organic Air Pollutants by Gas Chromatography and Mass Spectroscopy. EPA-600/2-79-057, 243 pp., March, 1979.

Protocol Prepared, June, 1980

APPENDIX C

ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS IN MILK

ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS IN MILK

1.0 Principle of the Method

Milk samples are collected from nursing mothers and frozen until ready for analysis. An aliquot of the thawed sample is then extracted, cleaned up by Florisil column chromatography and analyzed by GC/MS/COMP.

The extraction procedure used here is preferable to that used by the AOAC(1), since both polar and nonpolar compounds are extracted from the milk. The AOAC method is designed for pesticide residues and would not efficiently extract polar and/or acidic compounds.

Open column chromatography is a necessary prerequisite to GC/MS/COMP analysis. Although some loss of sample may occur during the extraction and cleanup, these procedures remove proteins and fats from the sample which would otherwise create overwhelming interferences for GC/MS/COMP analysis.

Since the compounds of interest in these fractions cover such a broad range of volatilities, the GC/MS/COMP analysis can be rather complex. The higher PBBs of interest in the extracted fraction must be chromatographed on a very short column (45 cm x 0.2-cm i.d., 2 percent OV-101 on Gas-Chrom Q) at high temperatures to elute them as sharp peaks which may be identified and quantitated. These chromatographic conditions are not applicable to more volatile compounds since they are not resolved from the solvent. Thus, the extracted fraction is analyzed a second time using a nonpolar SCOT capillary column (either OV-101 or SE-30 liquid phase) to separate and identify semivolatile constituents (e.g. chlorobenzenes, PCNs, pesticides, etc.). The chromatographic conditions are typically 60°C initially, programmed to 240°C (or the column limit) at 6°/min.

The mass spectral data are stored on magnetic tape. The mass spectra of interest will be printed out by the instrument operator for qualitative analysis. Quantitation from this data may be achieved by integrating the area of selected ions and comparing them to the area of the external standard.

The sensitivity of the determination may be significantly improved for quantitative purposes by using the technique of selected ion monitoring (SIM), also known as multiple ion detection (MID). This technique monitors up to 9 ions at a sensitivity 10-100 greater than the normal operating mode. This technique is used for quantitation of compounds in samples where the increased sensitivity is necessary for detection or accurate determination.

2.0 Range and Sensitivity

The detection limit of the GC/MS/COMP system has been determined to be about 5-50 ng/μL for pesticides such as γ-BHC, p,p'-DDE, atrazine, trifluralin and heptachlor using a 40 m SE-30 capillary column. When SIM was used, the detection limit was about one order of magnitude less (i.e., 0.5-5 ng/μL). The detection limit for tetrabromobiphenyl is about 1 ng/μL in the SIM mode using 45 x 0.2-cm i.d. column packed with 2 percent OV-101 coated on Gas-Chrom Q.

For an instrumental detection limit of 1 ng/μL, the overall sensitivity of the method should be about 6 ng/mL (6 ppb) milk assuming a 50 mL milk sample extracted and extract concentrated to 0.3 mL. This detection limit may be improved by using SIM and may be worsened by background interferences.

3.0 Precision and Accuracy

When electron capture gas chromatography (GC/ECD) was used, the mean recoveries from cow's milk for seven replicates ranged from 57 to 93 percent for six model compounds. Thus, the results obtained may be as little as half the actual amount in the sample. The relative standard deviations (RSD) for the above replicates ranged from 11 to 33 percent, with the average RSD at 21.7 percent. Thus the precision of the method is about ± 20 percent. It is anticipated that accuracy and precision will improve with experience with the method.

4.0 Apparatus

4.1 Gas Chromatograph

A Fisher-Victoreen 4400 gas chromatograph with an ^{3}H electron capture detector, a 10^{-13} AFS electrometer, and a 1.0 mV recorder is used.

4.2 Gas Chromatography Column

For most compounds, separation is achieved using a 40 m SCOT glass capillary column coated with 1 percent SE-30 and 0.32 percent Tullanox. For the compounds of very low volatility (e.g. the higher PBBs) which will not chromatograph on the capillary column, a 45- x 0.2-cm i.d. glass column packed with 2 percent OV-101 on Gas-Chrom Q is used.

4.3 Liquid Chromatography Column

A 24-mm i.d. glass column with a Teflon stopcock is used.

4.4 Gas Chromatography/Mass Spectrometer

An LKB 2091 gas chromatograph/mass spectrometer with 2 PDP 11/4 computer is used. The system is equipped with a glass jet separator and is used with either glass capillary or packed glass column.

5.0 Materials

Kuderna-Danish evaporators:
  5 mL receivers
  250 mL KD flasks
  Snyder columns
  500 mL flat-bottom boiling flasks
250 mL separatory funnels
Clean glass wool
Whatman 1 P/S filter paper
Florisil
Sodium sulfate (anhydrous)
Acetone "Distilled in Glass", redistilled
Pentane "Distilled in Glass", redistilled
Toluene "Distilled in Glass", redistilled
Ethyl ether "Distilled in Glass"

6.0 Procedure

6.1 Extraction

(1) Mix 50 mL (or volume available up to 50 mL) of a milk sample with clean glass wool and 150 mL of acetone to precipitate the proteins.

(2) Decant and filter the acetone/water layer.

(3) Repeat steps 1 and 2 with two 50 mL acetone fractions.

(4) Concentrate to about 20 mL using a Kuderna-Danish evaporator.

(5) Extract the precipitate with 40 mL of toluene; decant and filter the toluene layer.

(6) Combine the toluene extract and the acetone extract with shaking.

(7) Let the layers separate and draw off toluene (top) layer.

(8) Repeat Steps 5-7 with 40 mL toluene and then with 10-20 mL toluene.

(9) Discard the lower water layer.

(10) Dry the organic layer with anhydrous sodium sulfate and concentrate to desired volume using a flat-bottom boiling flask and Snyder column. Quantatively transfer to a vial and concentrate to 5-10 mL under a gentle stream of nitrogen.

6.2 Florisil Column Chromatography(1)

(1) Prepare Florisil by heating to 130°C for at least 5 hours.

(2) Prepare a 24-mm i.d. column so that the Florisil is 10 cm high after settling.

(3) Place about 1 cm of anhydrous sodium sulfate on top of the Florisil.

(4) Rinse column with 40-50 mL pentane, never allowing the solvent to go below the Na_2SO_4 layer, as channeling may result.

(5) Add up to 10 mL of sample to column.

(6) Elute with 200 mL of 6 percent ethyl ether/pentane solution at ≤ 5 mL/min.

(7) Collect and concentrate in a Kuderna-Danish evaporator.

(8) Evaporate under nitrogen stream to ~ 1.5 mL. Quantitatively transfer to a vial, store in a freezer.

(9) If sample solidifies after concentration, repeat the Florisil cleanup (Steps 1-8).

6.3 Standards

Standards are spiked into the sample following the extraction and workup (d_{10}-pyrene was used at 200 ng/mL).

6.4 Analysis

6.4.1 GC/MS/COMP Analysis for Semivolatiles

Inject 0.2 μL onto a 40 m SE-30 SCOT capillary at 60°C initially, program at 6°/min to 240°C, then hold until no more peaks are observed. Collect mass spectral data at 2 sec/scan from m/z 20-500. Compounds amenable to this analysis include organic compounds with volatility lower than that for purgeable compounds. Only the very low volatile compounds (e.g. higher PBBs) will not elute from the capillary.

6.4.2 GC/MS/COMP Analysis for Low Volatile Compounds

6.4.2.1 Normal Procedure

Inject 1.0 μL onto a 45 x 0.2-cm i.d. glass column packed with 2 percent OV-101 on GasChrom Q at 220°C initially, program to 300° at 12°/min and hold until all peaks have eluted. A helium flow rate of 20 mL/min is used. The mass spectrometer is scanned from *m/z* 20-1000 at 2 sec/scan.

6.4.2.2 Alternate Procedure

Using the same chromatographic conditions analyze the sample by SIM. Preselect up to 8 ions characteristic of the compound(s) of interest and one ion characteristic of the standard. Retention times provide qualitative identifications. Peak areas may be used for quantification as discussed below. This alternate procedure has 10-100 times better sensitivity than the full scan mode and provides faster quantitative results. The main disadvantage is that only preselected compounds may be identified.

In addition, if specific halogenated compounds are found to be present with little interference in most samples, they may be analyzed by GC/ECD. This procedure improves the sensitivity and reduces the analysis time (since GC/MS/COMP requires an offline data output). If GC/ECD is used, approximately 10 percent of the analyses are verified by GC/MS/COMP.

6.4.3 Qualitative Data Interpretation

Spectra are interpreted by visual comparison with standard spectral reference collections(2,3) where possible. Where standard spectra are not available, tentative identifications are made based upon interpretation of the mass spectrum. Where possible, the GC retention time is also used to assist in the identification procedure.

All identifications and interpretations are checked independently by other experienced chemists or spectroscopists to assure that the interpretations are correct.

6.4.4 Quantitative Analysis

In order to eliminate the need to construct complete calibration curves for each compound to be quantified, the method of relative molar response (RMR) is used. Successful use of this method requires information on the exact amount of standard added and the relationship of RMR (unknown) to the RMR (standards). In general, the RMR for a compound is determined for a characteristic ion (parent or fragment) in its mass spectrum. The integrated ion current may also be used, but is generally less precise. The value of RMR is determined from at least three independent analyses. The method of calculation is as follows:

$$(1)\quad RMR_{unknown/standard} = \frac{A_{unk}/moles_{unk}}{A_{std}/moles_{std}}$$

A = peak area, determined by integration or triangulation of the total ion current or for a selected mass of each compound

$$(2)\quad RMR_{unk/std} = \frac{A_{unk}/g_{unk}/GMW_{unk}}{A_{std}/g_{std}/GMW_{std}}$$

A = peak area, as above
g = number of grams present
GMW = gram molecular weight

Thus, in the sample analyzed:

$$(3)\quad g_{unk} = \frac{A_{unk}/GMW_{unk}/g_{std}}{A_{std}/GMW_{std}/RMR_{unk/std}}$$

7.0 References

1. Horowitz, W., ed., AOAC Methods of Analysis, 12th ed., Association of Official Analytical Chemists, Washington, DC. (1975).
2. McLafferty, F. W., E. Stenhagen, and S. Abrahammson, ed., "Registry of Mass Spectral Data," John Wiley and Sons, New York (1974).
3. Eight Peak Index of Mass Spectra. Vol. I (Tables 1 and 2) and II (Table 3), Mass Spectrometry Data Centre, AWRE, Aldermaston, Reading, RG74PR, UK (1970).

Protocol Prepared, June, 1980

APPENDIX D

VOLATILE COMPOUNDS IDENTIFIED IN SELECTED PURGES OF MOTHER'S MILK

Table D-1. VOLATILE COMPOUNDS IDENTIFIED IN PURGE OF SAMPLE NO. 1081 (Bayonne, NJ)

Chromato- graphic Peak No.	Elution Temp. (°C)	Compound
1A	58	carbon dioxide
1B	58	chlorotrifluoromethane
2	61	propylene
3	65	C_4H_8 isomer
4	66	C_4H_{10} isomer
5	67	C_4H_8 isomer
6	73	acetaldehyde
7A	73	acetone
7B	74	trichlorofluoromethane
8	76	n-pentane
9	77	isopropanol
10	79	methylene chloride
11	80	freon 113
12	83	carbon disulfide
13	83	n-butanal
14	87	cyclopentane
15	89	$C_4H_6O_2$ isomer
16	91	methyl ethyl ketone
17	92	C_6H_{12} isomer
18	94	hexafluorobenzene (int. std.)
19	95	n-hexane
20	96	chloroform
21	97	C_7H_{14} isomer
22	99	C_6H_{12} isomer
23A	102	perfluorotoluene (int. std.)
23B	102	methylcyclopentane
24	104	1,1,1-trichloroethane
25	105	C_7H_{14} isomer
26	108	benzene
27	112	cyclohexane
28A	113	ethyl vinyl ketone
28B	114	2-pentanone
29	115	$C_6H_{12}O$ (tent.)
30	116	n-pentanal
31A	119	trichloroethylene
31B	119	C_7H_{12} or C_6H_8O isomer
32	122	n-heptane
33	126	C_8H_{16} isomer
34	129	C_7H_{14} isomer
35	134	1-chloropentane
36	135	unknown
37	138	toluene
38	143	$C_6H_{12}O$ isomer (tent.)
39	145	n-hexanal
40	147	C_8H_{16} isomer
41	150	n-octane
42A	152	tetrachloroethylene
42B	152	C_8H_{16} isomer (tent.)
43	154	C_8H_{16} isomer (tent.)
44	156	siloxane
45	159	C_8H_{16} isomer (tent.)
46	161	chlorobenzene
47	163	1-chlorohexane (tent.)
48	166	ethylbenzene
49	168	xylene isomer
50	171	3-heptanone
51	171	2-heptanone
52	173	styrene
53A	173	C_9H_{16} isomer
53B	173	C_9H_{20} isomer
53C	174	n-heptanal
53D	174	xylene isomer
54	175	$C_{10}H_{22}$ isomer (tent.)
55	178	n-nonane
56	179	$C_{10}H_{22}$ isomer
57	181	3-methyl-1-iodobutane
58A	183	isopropylbenzene
58B	184	$C_{10}H_{22}$ isomer
59	188	$C_{11}H_{24}$ isomer
60A	189	$C_{10}H_{16}$ isomer
60B	189	$C_8H_{16}O$ isomer (tent.)
61A	191	benzaldehyde
61B	191	n-propyl benzene
62	193	C_3-alkyl benzene
63	194	C_9H_{20} isomer (tent.)
64	195	C_9H_{18} isomer
65	196	$C_{11}H_{24}$ isomer
66	197	octanone isomer
67	199	$C_{11}H_{24}$ isomer
68	200	2-pentylfuran
69A	201	$C_{11}H_{24}$ isomer
69B	202	n-octanal
70	203	siloxane
71A	204	$C_{10}H_{22}$ isomer
71B	205	dichlorobenzene
72	206	$C_{11}H_{24}$ isomer
73A	210	$C_{10}H_{14}$ isomer
73B	210	C_9H_{16} isomer (tent.)
73C	210	sat. hydrocarbon
74	211	sat. hydrocarbon

- Continued -

Table D-1 (cont'd.)

Chromatographic Peak No.	Elution Temp. (°C)	Compound
75	212	limonene
76	215	sat. hydrocarbon
77A	215	unsat. hydrocarbon
77B	216	$C_{11}H_{24}$ isomer (tent.)
78	218	monochlorodecane (tent.)
79A	219	$C_9H_{18}O$
79B	219	acetophenone
80	221	sat. hydrocarbon
81	222	sat. hydrocarbon
82	224	2-nonanone
83	225	dimethylstyrene
84	227	n-nonanal
85	230	n-undecane
86	240	unsat. hydrocarbon
87	240	siloxane
88	240	naphthalene
89	240	$C_{10}H_{20}O$ isomer (tent.)
90	240	n-dodecane
91	240	unknown
92	240	unsat. hydrocarbon
93	240	siloxane
94	240	$C_{11}H_{22}$ isomer
95	240	siloxane
96	240	unknown
97	240	siloxane

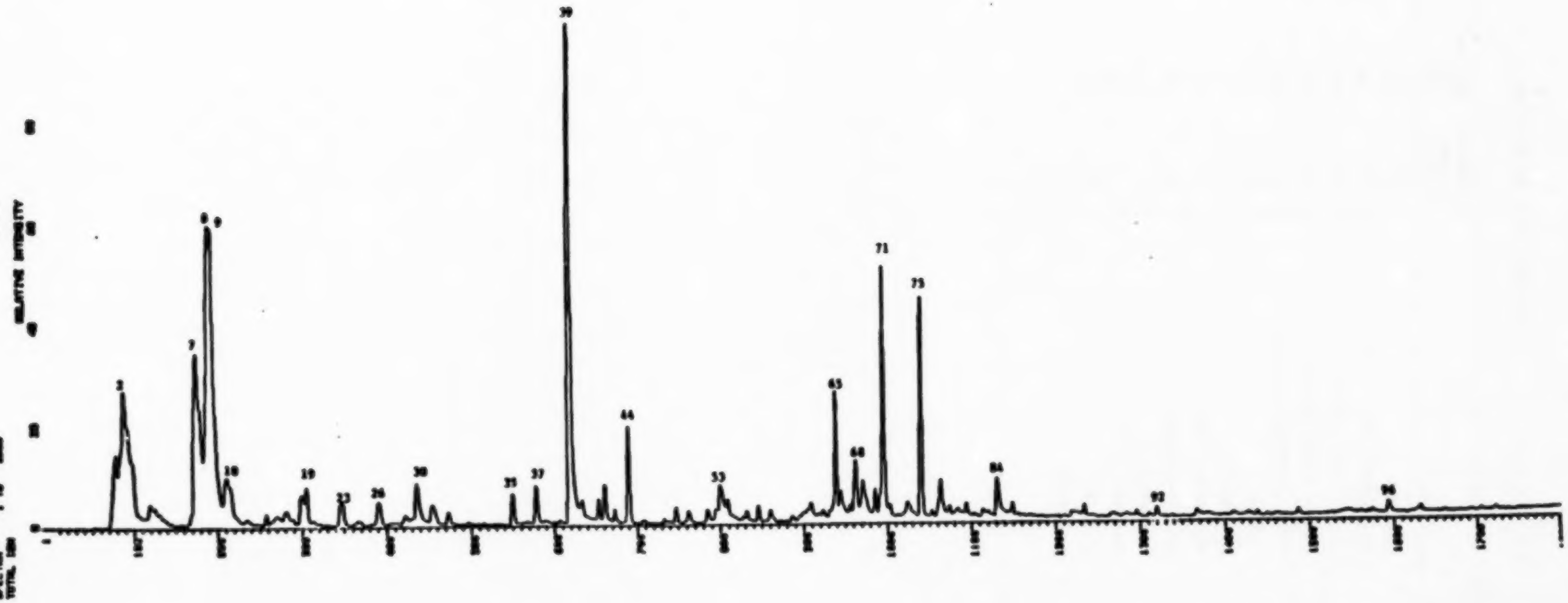

Figure D-1. Total ion current chromatogram from GC/MS analysis for volatiles in sample no. 1081 (Bayonne, NJ).

Table D-2. VOLATILE COMPOUNDS IDENTIFIED IN PURGE OF SAMPLE NO. 1040 (Bayonne, NJ)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
1	58	carbon dioxide
2	59	chlorotrifluoromethane
3	60	dimethyl ether
4	67	C_4H_{10} isomer
5A	74	isopentane
5B	74	trichlorofluoromethane
5C	75	acetone
5D	75	C_5H_{10} isomer
6A	77	*n*-pentane
6B	78	isoprene
6C	78	isopropanol
6D	79	C_6H_{12} isomer
6E	79	vinylidine chloride
7	81	methylene chloride
8	82	Freon 113
9	84	carbon disulfide
10	85	2-methylpropanal
11	87	cyclopentane
12	90	unknown
13	92	methyl ethyl ketone
14	94	C_6H_{12} isomer
15	96	hexafluorobenzene (int. std.)
16	97	*n*-hexane
17	98	chloroform
18	101	C_6H_{12} isomer
19	104	perfluorotoluene (int. std.)
20A	106	1,1,1-trichloroethane
20B	107	3-methylbutanal (tent.)
21	109	2-methylbutanal
22	110	benzene
23	111	carbon tetrachloride
24A	113	cyclohexane
24B	113	methyltetrahydrofuran (tent.)
25A	113	C_7H_{14}
25B	115	ethyl vinyl ketone
26	115	2-pentanone
27A	117	vinyl propionate (tent.)
28B	121	trichloroethylene
28A	123	C_7H_{12} or C_6H_8O
28B	124	unknown
29	127	C_7H_{14} isomer
30	130	C_7H_{14} isomer
31	132	dimethyl disulfide
32	136	1-chloropentane
33	138	unknown
34	140	toluene
35A	141	1-pentanol
35B	142	unknown
36	145	C_7H_{16} isomer
37	146	*n*-hexanal
38	149	C_8H_{16} isomer
39A	150	unknown
39B	151	C_8H_{16} isomer
40A	152	C_8H_{18} isomer
40B	153	trans-4-octene
41	153	tetrachloroethylene
42A	154	C_9H_{20} isomer
42B	154	sat. hydrocarbon
42C	154	unsat. hydrocarbon
43	155	C_8H_{16} isomer
44A	157	C_8H_{14} isomer
44B	157	siloxane
45	161	unsat. hydrocarbon
46A	162	sat. hydrocarbon
46B	162	unsat. hydrocarbon
47	163	unknown
48	165	chlorohexane
49	167	ethylbenzene
50	169	xylene isomer
51	173	2-heptanone
52A	174	styrene
52B	175	2-*n*-butylfuran (tent.)
53A	175	*n*-heptanal
53B	176	xylene isomer
54	177	C_9H_{18} isomer
55	179	C_9H_{20} isomer
56	181	sat. hydrocarbon
57	181	C_9H_{18} isomer
58A	182	3-methyl-1-iodobutane
58B	183	C_9H_{18} isomer
59A	184	isopropylbenzene
59B	185	sat. hydrocarbon
60	189	hydrocarbon
61	190	$C_{10}H_{16}$ isomer
62	190	unsat. hydrocarbon
63	191	benzaldehyde
64	192	*n*-propylbenzene (tent.)
65	194	trimethylbenzene isomer
66	196	isoamyl formate (tent.)
67A	196	unknown

- Continued -

Table D-2 (cont'd.)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
67B	197	sat. hydrocarbon
68A	198	C_9H_{20} isomer
68B	199	C_3-alkyl benzene
69	200	sat. hydrocarbon
70	201	2-pentyl furan
71	203	C_3-alkyl benzene
72	203	$C_{10}H_{20}$
73	204	siloxane
74	206	dichlorobenzene
75	207	C_3-alkyl benzene (tent.)
76	209	C_8H_{14}
77	211	dimethylethylbenzene isomer
78	212	menthene (tent.)
79	213	limonene
80	216	$C_{11}H_{22}$ isomer
81	216	unsat. hydrocarbon
82	217	sat. hydrocarbon
83	219	unknown
84	220	unknown
85	222	acetophenone
86	223	sat. hydrocarbon
87	225	$C_{10}H_{22}$ isomer
88	226	dimethylstyrene
89	228	n-nonanal
90A	230	siloxane
90B	231	siloxane
91	234	tetramethylbenzene (tent.)
92	239	siloxane
93	240	siloxane
94	240	naphthalene
95	240	$C_{12}H_{26}$ isomer
96	240	unknown
97	240	siloxane
98	240	2-undecanone
99	240	$C_{13}H_{28}$
100	240	siloxane

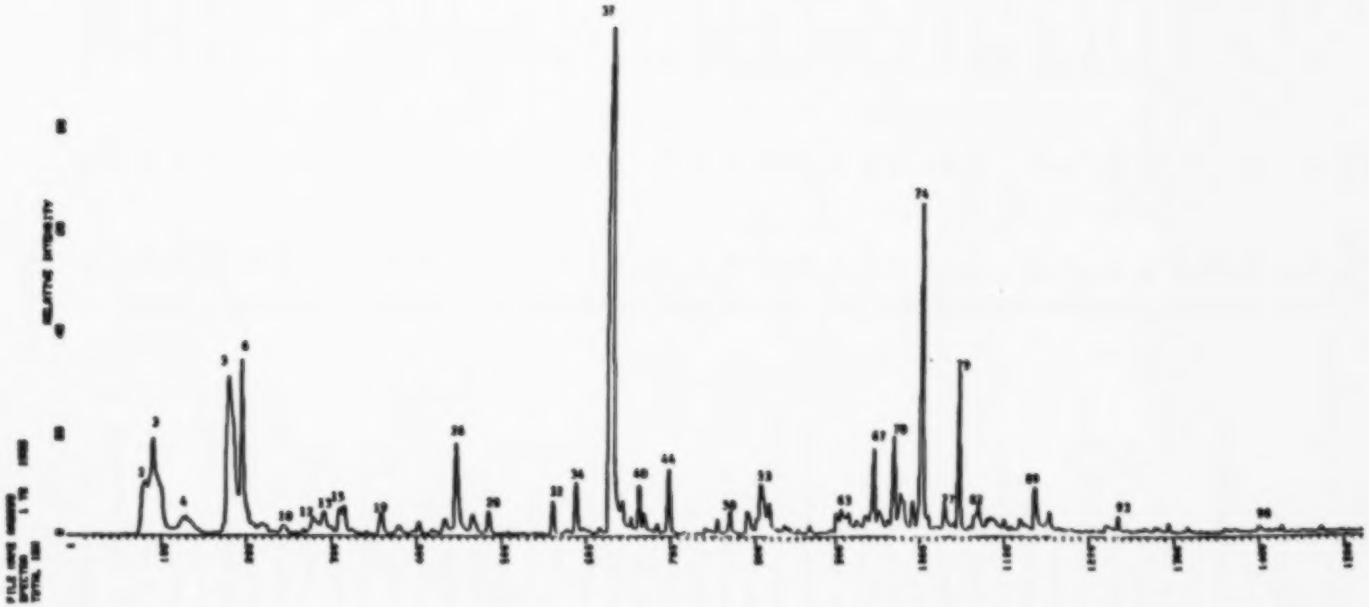

Figure D-2. Total ion current chromatogram from GC/MS analysis for volatiles in sample no. 1040 (Bayonnc, NJ).

Table D-3. VOLATILE COMPOUNDS IDENTIFIED IN PURGE OF SAMPLE NO. 1107 (Jersey City, NJ)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
1	64	xenon
2	65	carbon dioxide
3A	67	freon 22
3B	67	dichlorodifluoromethane
4	69	*n*-propane
5A	70	butene isomer
5B	71	*n*-butane
5C	72	acetaldehyde
5D	73	butene isomer
6A	74	chloroethane
7	75	tetramethylsilane
8A	76	trichlorofluoromethane
8B	78	1-pentene
8C	78	acetone
9A	79	isopropanol
9B	79	*n*-pentane
10A	81	methylene chloride
10B	83	Freon 113
10C	85	carbon disulfide (trace)
10D	86	methyl vinyl ketone (trace)
10E	86	methyl propanol
10F	86	nitromethane (tent.)
11A	88	cyclopentane
11B	89	2-methyl pentane
12A	90	vinyl acetate
12B	91	*n*-butanal
13A	92	3-methyl pentane
13B	93	C_6H_{12} isomer
14A	94	perfluorobenzene (int. std.)
14B	97	*n*-hexane
14C	98	chloroform
15	100	dihydrofuran
16A	101	tetrahydrofuran
16B	102	perfluorotoluene (int. std.)
16C	102	methylcyclopentane
17A	104	*n*-methyl acetamide
17B	105	1,1,1-trichloroethane
17C	106	3,3-dimethyloxetan (tent.)
18A	108	benzene
18B	109	carbon tetrachloride
19A	110	1-butanol
19B	110	cyclohexane
19C	111	$C_5H_{10}O$ isomer
19D	112	ethyl vinyl ketone (tent.)
20A	112	2-pentanone
20B	113	vinyl propionate
21	114	*n*-pentanal
22A	116	C_7H_{14} isomer
22B	117	trichloroethylene
22C	118	*p*-dioxane
22D	118	ethyl furan (tent.)
23	120	*n*-heptane
24	123	2,2,4-trimethyl-1-pentene
25	124	isohexenal
26A	125	$C_6H_{10}O$ isomer
26B	127	4-methyl-2-pentanone
26C	127	C_8H_{16} isomer
27	128	dimethyl disulfide
28	129	dihydropyran
29	131	chloropentane
30A	134	toluene
30B	137	C_8H_{18} isomer
31	139	$C_6H_{12}O$ isomer
32A	141	*n*-hexanal
32B	143	C_8H_{16} isomer
33A	146	*n*-octane
33B	147	C_8H_{16} isomer
34	148	tetrachloroethylene
35	149	C_8H_{16} isomer
36	151	siloxane
37	154	unknown
38A	156	C_9H_{18} isomer
38B	156	chlorobenzene
38C	156	2-hexenal (tent.)
39A	158	chlorohexane
39B	159	$C_7H_{12}O$ isomer
40A	160	ethyl benzene
40B	161	C_9H_{18} isomer
40C	161	4-heptanone
41A	162	xylene isomer
41B	163	phenylacetylene
42A	164	3-heptanone
42B	165	[illegible]ptanone
43	166	$C_7H_{12}O$ (tent.)
44A	167	styrene
44B	168	*n*-heptanal
44C	168	xylene isomer
45A	169	sat. hydrocarbon
45B	170	C_9H_{18} isomer
46	172	*n*-nonane

- Continued -

Table D-3 (cont'd.)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
47	173	sat. hydrocarbon
48	174	$C_{10}H_{20}$ isomer
49A	175	sat. hydrocarbon
49B	175	ethyl methyl cyclohexane
49C	176	unknown
49D	176	$C_7H_{10}O$ isomer
50A	176	isopropyl benzene
50B	177	$C_{10}H_{22}$ isomer
51	178	$C_8H_{14}O$ isomer (tent.)
52A	181	trans-2-heptenal
52B	182	α-pinene
52C	182	benzaldehyde
53	184	*n*-propylbenzene
54	186	xylene isomer
55A	187	sat. hydrocarbon
55B	187	$C_{10}H_{22}$ isomer
56	187	benzonitrile (trace)
57	188	sat. hydrocarbon
58A	190	phenol
58B	190	trimethylbenzene
59	192	pentyl furan
60A	193	*n*-octanal
60B	193	benzofuran
61A	194	trimethylbenzene isomers
61B	194	$C_{10}H_{20}$ isomer
62	195	siloxane
63A	196	$C_7H_{10}O$
63B	196	*n*-decane
63C	197	dichlorobenzene
63D	198	$C_{11}H_{22}$ isomer
64A	200	unknown
64B	200	trimethyl benzene isomer
64C	201	unknown
64D	201	C_4-alkylbenzene
64E	201	sat. hydrocarbon
65	202	$C_{11}H_{25}$ isomer
66A	203	sat. hydrocarbon
66B	203	limonene
66C	204	$C_{11}H_{22}$ isomer
66D	204	methyl styrene
67A	205	sat. hydrocarbon
67B	206	$C_{11}H_{22}$ isomer
67C	206	diethylbenzene isomer
68A	207	sat. hydrocarbon
68B	207	acetophenone
69A	208	C_4-alkylbenzene
69B	209	$C_{11}H_{22}$ isomer
70	210	$C_{11}H_{22}$ isomer
71A	210	$C_{11}H_{22}$ isomer
71B	211	phthalide (tent.)
72A	211	sat. hydrocarbon
72B	212	decalin (tent.)
73A	212	sat. hydrocarbon
73B	212	$C_{11}H_{24}$ isomer
73C	213	C_4-alkylbenzene isomer
74	213	2-nonanone
75A	214	$C_{11}H_{22}$ isomer
75B	215	C_4-alkyl benzene isomer
76A	215	sat. hydrocarbon
76B	216	*n*-nonanal
77	217	$C_{11}H_{22}$ isomer
78A	218	$C_{10}H_{12}O$ isomer
78B	219	*n*-undecane
79A	220	siloxane
79B	220	$C_{11}H_{22}$ isomer
80	221	$C_{10}H_{18}$ isomer
81	222	C_4-alkylbenzene isomer
82A	223	$C_{12}H_{26}$ isomer
82B	224	$C_{12}H_{24}$ isomer
83	224	2-methyldecalin (tent.)
84A	225	$C_{12}H_{26}$
84B	226	C_5-alkylbenzene isomer
84C	226	C_4-alkylbenzene isomer
85	226	siloxane (tent.)
86A	228	$C_{12}H_{24}$ isomer
86B	228	$C_{11}H_{20}$ isomer (trace)
86C	229	$C_{12}H_{24}$ isomer
86D	229	$C_{10}H_{12}O$ isomer
86E	229	$C_{10}H_{18}O$ isomer
86F	230	unknown
86G	230	$C_{11}H_{16}$ isomer
87	230	siloxane
88	230	sat. hydrocarbon
89	230	sat. hydrocarbon
90	230	sat. hydrocarbon
91	230	sat. hydrocarbon
92	230	naphthalene
93	230	unsat. hydrocarbon
94	230	*n*-dodecane
95	230	sat. hydrocarbon

- Continued -

Table D-3 (cont'd.)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound	Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
96	230	siloxane	105	230	unknown
97	230	2-undecanone	106	230	2-tridecanone
98	230	sat. hydrocarbon	107	230	sat. hydrocarbon
99	230	unknown	108	230	siloxane
100	230	siloxane	109	230	phthalate
101	230	sat. hydrocarbon	110	230	lactone isomer (tent.)
102	230	unknown	111	230	diisobutyrate isomer
103	230	diphenyl ether	112	230	$C_{14}H_{22}O$ isomer
104	230	sat. hydrocarbon			

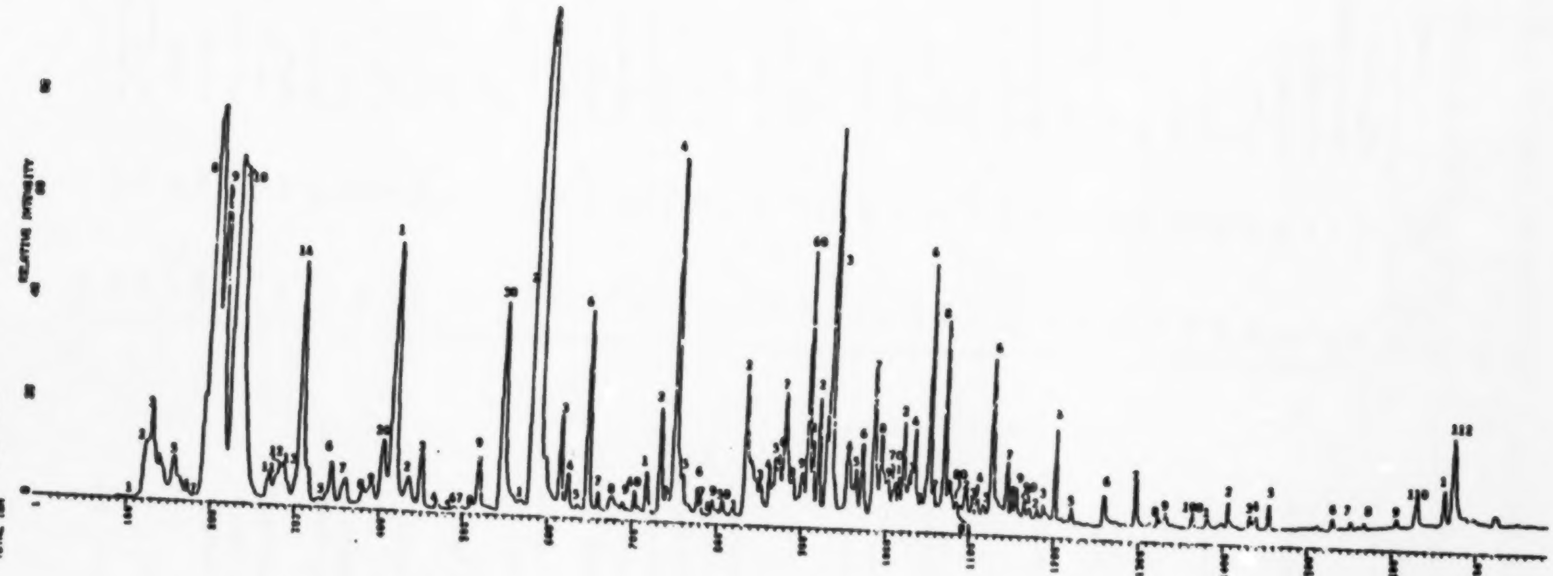

Figure D-3. Total ion current chromatogram from GC/MS analysis for volatiles in sample no. 1107. (Jersey City, NJ).

Table D-4. VOLATILE COMPOUNDS IDENTIFIED IN PURGE OF SAMPLE NO. 1115 (Jersey City, NJ)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
1A	62	carbon dioxide
1B	63	xenon (trace)
2	65	carbonyl sulfide (tent.)
3A	67	chloromethane
3B	68	unknown
4A	76	trichlorofluoromethane
4B	76	acetone
5A	77	isopentane
5B	78	isopropanol
6A	80	methylene chloride
6B	81	Freon 113
6C	82	carbon disulfide (trace)
6D	82	unknown
7	83	unknown
8A	86	cyclopentane
8B	87	methyl isopropyl ketone
8C	89	n-butanal
9	90	1-hexene (tent.)
10A	92	hexafluorobenzene (int. std.)
10B	92	n-hexane
11A	94	chloroform (trace)
11B	94	methyl furan
12	96	unsat. hydrocarbon
13	98	perfluorotoluene (int. std.)
14A	99	crotonaldehyde (tent.)
14B	100	1,1,1-trichloroethane
14C	100	3-methylbutanal
15	102	2-methylbutanal (tent.)
16A	104	benzene
16B	105	carbon tetrachloride (trace)
16C	105	1-butanol (tent.)
17	106	unknown
18A	107	ethyl vinyl ketone
18B	107	2-pentanone
19	108	vinyl propionate
20A	109	n-pentanal
20B	110	sat. hydrocarbon
20C	110	methylhexane (tent.) (trace)
21A	111	1-hexene
21B	112	trichloroethylene
21C	112	ethylfuran (tent.)
22A	114	2,5-dimethylfuran
22B	114	n-heptane
22C	115	C_6H_8 isomer
23A	116	unknown
23B	117	$C_5H_6N_2$ (tent.) (trace)
24	120	C_7H_{14} isomer
25	122	dimethyldisulfide
26	122	dihydropyran
27	124	chloropentane
28	126	unknown
29A	128	toluene
29B	129	1-pentanol
30	131	4-methyl-2-pentanone
31	134	n-hexanal
32A	136	C_8H_{16} isomer
32B	137	furaldehyde (tent.) (trace)
33	138	n-octane
34A	140	tetrachloroethylene
34B	140	dichloropropene (trace)
34C	141	unknown
35A	142	$C_5H_8N_2$
35B	142	C_8H_{16} isomer
36	143	siloxane
37A	146	2-hexanal
37B	147	chlorobenzene
38A	148	C_8H_{14} isomer
38B	149	5-methyl-3-hydrofuran-2-one (tent.)
39	151	α-furfuryl alcohol
40	151	ethylbenzene
41A	152	C_9H_{18} isomer
41B	152	$C_4H_4N_2O$ (tent.)
42A	153	xylene isomer
42B	153	phenylacetylene
42C	155	5-methyl-3-hexanone
43A	155	2-heptanone
43B	156	$C_7H_{12}O$
44A	157	C_9H_{20} (trace)
44B	158	styrene
44C	158	n-heptanal
44D	159	xylene isomer
45	159	C_9H_{18} isomer
46	160	2-furyl methyl ketone (tent.)
47	162	n-nonane
48	165	iodopentane
49	166	unknown
50	170	trans-2-heptenal
51A	171	benzaldehyde
51B	172	5-methyl-2-furfural
51C	172	unknown
51D	173	n-propylbenzene
52A	174	xylene isomer

-Continued-

Table D-4 (cont'd.)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
52B	175	benzonitrile
52C	175	octanone
52D	175	$C_{10}H_{22}$
52E	176	C_3-alkylbenzene
53A	176	1-chloro-3-ethylbenzene (tent.)
53B	176	dibromodichloromethane (tent.)
53C	176	phenol
53D	177	sat. hydrocarbon
53E	177	5-methyl-3-heptanone (tent.)
53F	177	unknown
54	178	6-methyl-2-heptanone
55	180	pentyl furan
56	180	*n*-octanal
57A	181	benzofuran (trace)
57B	182	C_3-alkylbenzene
57C	182	$C_{10}H_{20}$ isomer
57D	182	$C_7H_{10}O$ isomer
58	182	siloxane
59	184	*n*-decane
60	184	dichlorobenzene
61	187	C_9H_{16}
62A	188	C_4-alkylbenzene
62B	188	phenylacetaldehyde
62C	188	$C_{10}H_{20}$ isomer
63A	190	limonene
63B	190	1,8-cineole
63C	191	$C_{10}H_{18}$ (trace)
64	192	unsat. hydrocarbon
65A	192	sat. hydrocarbon
65B	193	acetophenone
66A	194	*n*-butylbenzene (tent.)
66B	195	$C_7H_8O_2$ (tent.)
67	196	$C_{11}H_{22}$ isomer
68	196	unknown
69	197	unknown
70A	198	$C_{10}H_{18}$ isomer
70B	198	sat. hydrocarbon
71A	199	2-nonanone
71B	200	dimethylstyrene (trace)
71C	200	C_4-alkylbenzene (trace)
71D	200	$C_{10}H_{16}O$ isomer
72	201	*n*-nonanal
73	204	undecane
74	212	unsat. hydrocarbon
75	213	$C_{10}H_{18}O$ isomer
76A	214	*n*-pentylbenzene
76B	215	siloxane
77	216	sat. hydrocarbon
78	218	2-decanone
79A	220	naphthalene
79B	220	$C_{12}H_{22}$ isomer
80	221	*n*-decanal
81	223	*n*-dodecane
82	225	sat. hydrocarbon
83A	226	unknown
83B	227	methyl cinnoline (tent.) (trace)
84	228	lactone isomer (tent.)
85	231	oxygenated hydrocarbon
86	233	phenyl hexane
87	237	$C_{10}H_{16}O$ (tent.)
88	238	unknown
89	239	undecane
90	240	$C_{10}H_{16}O$ (tent.)
91A	240	unknown
91B	240	siloxane
92	240	unsat. hydrocarbon
93	240	sat. hydrocarbon
94	240	2,2,4-trimethylpenta-1,3-diol di-isobutyrate (BKG)
95	240	sat. hydrocarbon
96	240	$C_{14}H_{30}$ isomer
97	240	unsat. hydrocarbon
98	240	sat. hydrocarbon
99	240	$C_{15}H_{32}$ isomer
100	240	sat. hydrocarbon

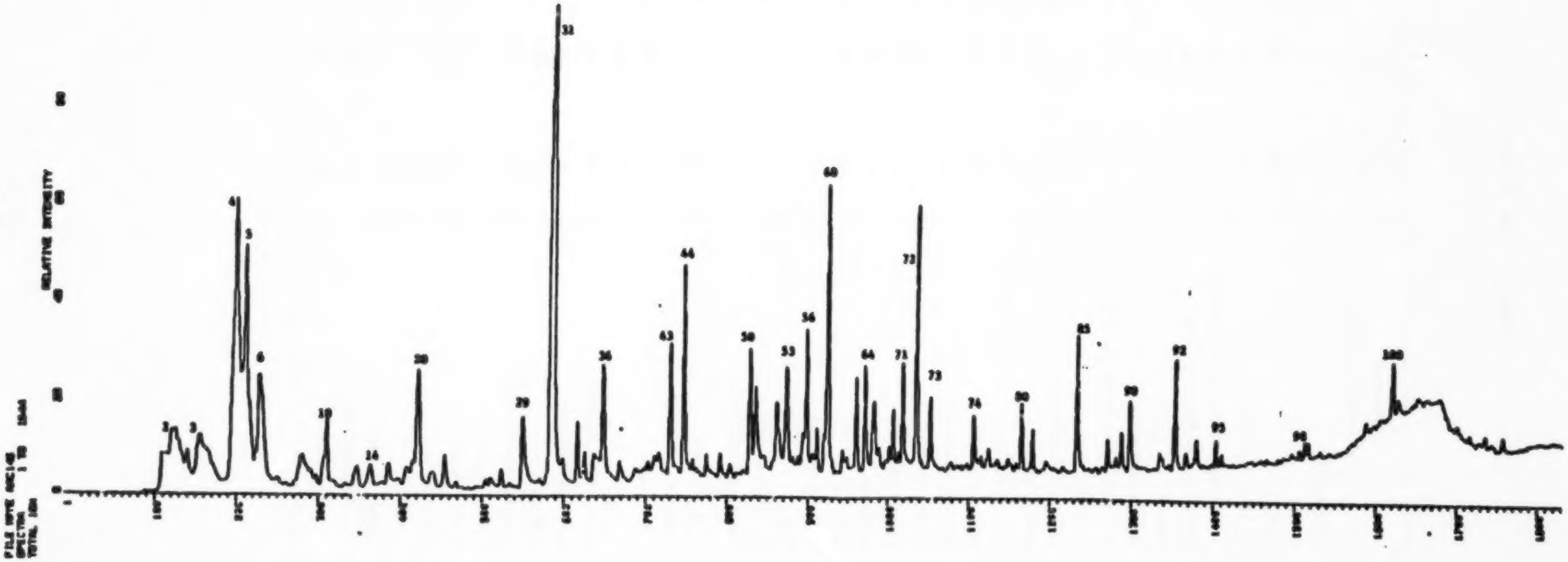

Figure D-4. Total ion current chromatogram from GC/MS analysis for volatiles in sample no. 1115 (Jersey City, NJ).

Table D-5. VOLATILE COMPOUNDS IDENTIFIED IN PURGE OF SAMPLE NO. 2048 (Pittsburgh, PA)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
1A	58	carbon dioxide
1B	58	chlorotrifluoromethane
2	64	C_4H_8 isomer
3	66	C_4H_{10} isomer
4A	70	acetaldehyde
4B	70	C_5H_{12} isomer
5A	71	trichlorofluoromethane
5B	72	acetone
6A	73	*n*-pentane
6B	74	isopropanol
7A	77	Freon 113
7B	77	methylene chloride
8	79	carbon disulfide
9A	83	C_5H_{10} isomer
9B	83	C_6H_{14} isomer
10	84	$C_5H_{10}O$ isomer (tent.)
11A	87	methyl ethyl ketone
11B	87	C_6H_{12} isomer
12A	89	hexafluorobenzene (int. std.)
12B	89	*n*-hexane
13	91	chloroform
14A	96	perfluorotoluene (int. std.)
14B	96	methylcyclopentane
15A	98	1,1,1-trichloroethane
15B	98	1-butanol (tent.)
16	102	benzene
17	104	cyclohexane
18A	106	C_6H_{12} isomer
18B	107	$C_5H_{10}O$ isomer
18C	109	C_6H_{10} isomer
19	109	*n*-pentanal
20A	112	trichloroethylene
20B	112	C_7H_{12} isomer
21	115	unknown
22	119	C_7H_{14}
23	126	$C_6H_{12}O$ isomer
24A	126	unsat. hydrocarbon
24B	127	chloropentane
25	130	unsat. hydrocarbon (tent.)
26	131	toluene
27	133	1-pentanol
28	134	C_6H_6 isomer
29	136	$C_6H_{12}O$ isomer
30	138	*n*-hexanal
31	140	C_8H_{16} isomer
32	143	*n*-octane
33A	145	tetrachloroethylene
33B	146	C_8H_{16} isomer
34	147	$C_7H_{14}O$ isomer
35	149	siloxane
36A	153	$C_6H_{12}O$ isomer
36B	154	chlorobenzene (trace)
37	156	chlorohexane (trace)
38	159	ethylbenzene
39A	161	sat. hydrocarbon
39B	161	xylene isomer
39C	162	unknown
39D	162	C_9H_{20} isomer
40	164	3-heptanone
41	165	2-heptanone
42A	166	styrene
42B	167	C_9H_{16} isomer (tent.)
42C	167	sat. hydrocarbon
43A	168	*n*-heptanal
43B	168	xylene isomer
44	169	*n*-nonane
45	170	$C_{10}H_{20}$ isomer
46	173	$C_{10}H_{22}$ isomer
47	175	$C_{10}H_{22}$ isomer (tent.)
48A	177	isopropylbenzene
48B	177	$C_{10}H_{22}$ isomer
49	181	$C_{11}H_{24}$ isomer
50A	182	$C_{10}H_{16}$ isomer
50B	183	$C_8H_{16}O$ isomer
51A	184	unsat. hydrocarbon
51B	184	benzaldehyde
51C	184	*n*-propylbenzene
52A	186	$C_{10}H_{16}$ isomer
52B	186	C_3-alkyl benzere isomer
53	187	sat. hydrocarbon
54	189	unsat. hydrocarbon
55	190	$C_{11}H_{24}$ isomer
56A	190	$C_8H_{16}O$ isomer
56B	192	$C_{10}H_{22}$ isomer
57	192	$C_{11}H_{24}$ isomer
58	194	2-pentylfuran
59	194	$C_{11}H_{24}$ isomer (tent.)
60A	195	C_3-alkylbenzene isomer
60B	195	$C_{10}H_{20}$ isomer
61	197	siloxane
62A	198	sat. hydrocarbon
62B	198	dichlorobenzene

-Continued-

Table D-5 (continued)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound	Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
63A	200	unsat. hydrocarbon	82	231	unsat. hydrocarbon
63B	200	sat. hydrocarbon (tent.)	83	232	$C_{11}H_{20}$ isomer
64	202	unsat. hydrocarbon	84	233	$C_{10}H_{18}O$ isomer
65	203	2-ethyl-1-hexanol	85A	235	siloxane
66A	206	limonene	85B	236	$C_{10}H_{18}O$ isomer
66B	206	$C_{10}H_{18}O$ isomer	85C	236	$C_{10}H_{14}O$ isomer
67	208	sat. hydrocarbon (tent.)	86	238	unsat. hydrocarbon
68	209	sat. hydrocarbon	87	240	sat. hydrocarbon
69	211	C_4-alkylbenzene	88A	240	naphthalene
70	212	acetophenone	88B	240	$C_{10}H_{22}O$ isomer (tent.)
71	213	sat. hydrocarbon	89A	240	α-terpineol (tent.)
72	214	sat. hydrocarbon	89B	240	unsat. hydrocarbon
73	215	sat. hydrocarbon	90	240	*n*-dodecane
74A	216	C_4-alkylbenzene	91	240	siloxane
74B	217	$C_9H_{18}O$ isomer	92	240	unsat. hydrocarbon
75A	218	dimethylstyrene	93	240	siloxane
75B	219	sat. hydrocarbon	94	240	2-undecanone
76	220	*n*-nonanal	95	240	siloxane
77	222	*n*-undecane	96	240	$C_{13}H_{28}$ isomer
78	223	siloxane	97	240	siloxane
79	226	C_4-alkylbenzene	98	240	decanoic acid (tent.)
80A	226	C_4-alkylbenzene	99	240	$C_{14}H_{30}$ isomer
80B	227	unknown	100	240	unsat. hydrocarbon
81	229	sat. hydrocarbon	101	240	siloxane

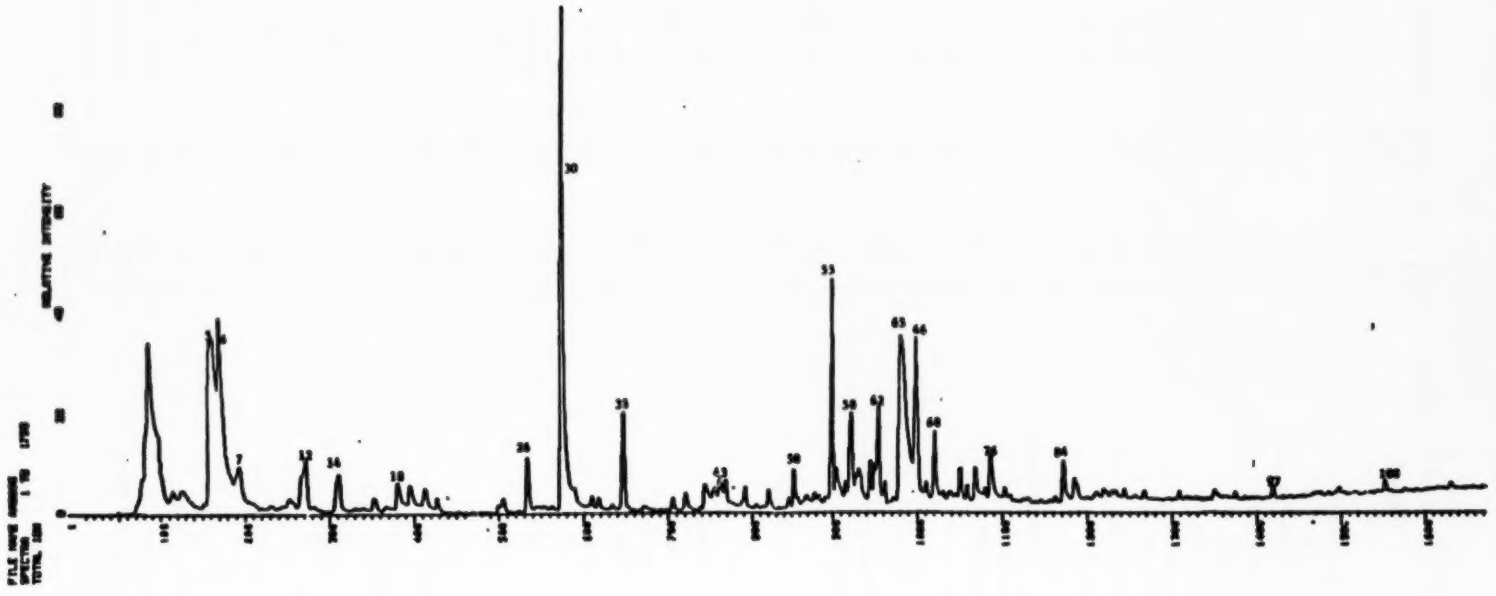

Figure D-5. Total ion current chromatogram from GC/MS analysis for volatiles in sample no. 2048 (Pittsburgh, PA).

Table D-6. VOLATILE COMPOUNDS IDENTIFIED IN PURGE OF SAMPLE NO. 2071 (Pittsburgh, PA)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
1	59	carbon dioxide
2A	60	propylene (trace)
2B	61	dichlorodifluoromethane (trace)
3A	62	dimethyldifluorosilane
3B	63	isobutane
4A	64	C_4H_8 isomer
4B	65	*n*-butane (trace)
5	66	acetaldehyde
6	68	chloroethane (trace)
7	71	methanol
8A	73	acetone
8B	73	trichlorofluoromethane
9A	75	isopropanol
9B	75	*n*-pentane
9C	76	C_5H_8 isomer
10	77	C_6H_{12} isomer
11A	78	methylene chloride
11B	79	2-methyl-2-propanol
11C	80	Freon 113
12	81	C_6H_{14}
13A	82	carbon disulfide
13B	83	C_4H_8O
14	85	*n*-propanol (tent.)
15A	86	cyclopentane
15B	87	C_6H_{12} isomer
16	87	C_6H_{14} isomer
17	88	vinyl acetate
18	89	*n*-butanal
19	90	methyl ethyl ketone
20	91	C_6H_{12} isomer
21	93	hexafluorobenzene (int. std.)
22	94	*n*-hexane
23A	94	ethyl acetate
23B	95	chloroform
24	96	C_7H_{14} isomer
25A	100	perfluorotoluene (int. std.)
25B	100	methylcyclopentane
26	101	C_7H_{14} isomer
27A	102	1,1,1-trichloroethane
27B	103	$C_5H_{10}O$ isomer (tent.)
28	106	benzene
29	107	carbon tetrachloride (trace)
30A	108	*n*-butanol (tent.)
30B	108	cyclohexane
31	111	methyl propyl ketone
32	113	*n*-pentanal
33	116	trichloroethylene
34A	118	*n*-heptane
34B	119	C_7H_{14} isomer
35	122	C_8H_{16} isomer
36	124	C_7H_{14} isomer
37	126	dimethyl disulfide
38	127	unknown
39	129	C_7H_{14} isomer (tent.)
40	133	toluene
41	138	dibromochloromethane (trace)
42	139	*n*-hexanal
43	141	C_8H_{16} isomer
44	144	*n*-octane
45A	145	tetrachloroethylene
45B	146	C_8H_{16} isomer (tent.)
46	147	unknown
47A	149	unsat. hydrocarbon
47B	149	siloxane
48	152	C_9H_{18} isomer
49	153	chlorobenzene
50A	158	ethylbenzene
50B	159	C_9H_{18} isomer
51A	160	xylene isomer
51B	160	phenylacetylene
52A	162	3-heptanone
52B	163	2-heptanone
53	164	styrene
54	166	xylene isomer
55	167	*n*-heptanal
56	169	*n*-nonane
57	170	$C_{10}H_{22}$ isomer
58A	173	isopropylbenzene
58B	174	$C_{10}H_{22}$ isomer
59	176	$C_{10}H_{16}$ isomer
60	177	$C_{10}H_{20}$ isomer
61	179	α-pinene
62A	180	benzaldehyde
62B	180	*n*-propylbenzene
63A	182	$C_{10}H_{16}$ isomer
63B	182	C_3-alkylbenzene
64	184	trimethylbenzene isomer
65	185	$C_{10}H_{22}$ isomer
66	185	benzonitrile
67A	186	methylheptanone isomer
67B	186	α-methylstyrene
68A	187	trimethylbenzene isomer

- continued -

Table D-6 (continued)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
68B	187	sat. hydrocarbon
69A	188	ethyl n-caproate
69B	188	pentylfuran (tent.)
70A	190	benzofuran (tent.)
70B	190	C_4-alkylbenzene
70C	190	trimethylbenzene isomer
70D	191	phenol (trace)
71	192	siloxane
72A	192	$C_{10}H_{22}$ isomer
72B	193	dichlorobenzene
72C	193	unknown
72D	194	$C_{10}H_{16}$ isomer
73	194	sat. hydrocarbon
74	196	$C_{10}H_{16}$ isomer (tent.)
75A	196	$C_{10}H_{16}$ isomer
75B	197	C_4-alkylbenzene
76	199	limonene
77	201	unknown
78	203	sat. hydrocarbon
79A	205	acetophenone
79B	205	$C_{10}H_{16}$ isomer
80	207	sat. hydrocarbon
81	208	unknown
82	210	2-nonanone
83	211	dimethylstyrene
84	211	sat. hydrocarbon
85	212	camphene (tent.)
86	214	siloxane
87	215	sat. hydrocarbon
88	216	methyl caprylate
89	222	siloxane
90	223	camphor
91	225	$C_{10}H_{18}O$ (trace) (tent.)
92	227	siloxane
93	230	trichlorobenzene (trace)
94A	231	ethyl caprylate
94B	232	naphthalene
95	235	n-dodecane
96	239	unsat. hydrocarbon (tent.)
97	240	siloxane
98A	240	2-undecanone
98B	240	sat. hydrocarbon
99	240	sat. hydrocarbon
100	240	methyl decanoate
101	240	siloxane
102	240	$C_{14}H_{30}$ (tent.)
103	240	ethyl decanoate
104	240	unsat. hydrocarbon

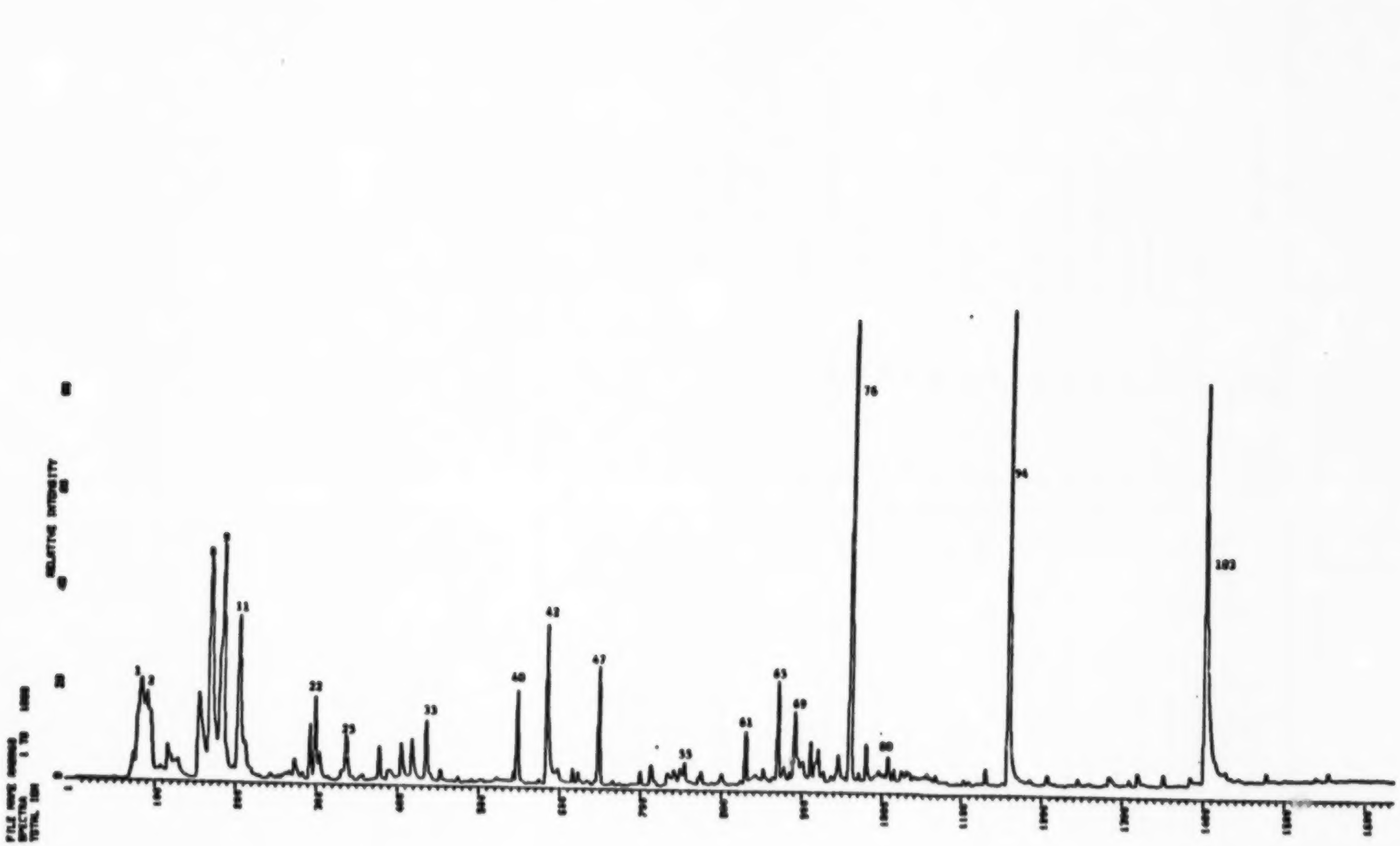

Figure D-6. Total ion current chromatogram from GC/MS analysis for volatiles in sample no. 2071 (Pittsburgh, PA).

Table D-7. VOLATILE COMPOUNDS IDENTIFIED IN PURGE OF SAMPLE NO. 3053 (Baton Rouge, LA)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound	Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
1A	37	carbon dioxide	34	136	$C_6H_{12}O$ isomer
1B	38	chlorotrifluoromethane	35	139	*n*-hexanal
2	62	chloromethane	36	141	C_8H_{16} isomer
3	63	C_4H_{10} isomer	37	144	*n*-octane
4	68	dimethyldifluorosilane	38	145	$C_6H_{10}O$ isomer
5A	70	acrylaldehyde	39	147	furaldehyde isomer
5B	71	acetone	40	149	C_8H_{16} isomer
5C	72	furan	41	150	siloxane
6	73	*n*-pentane	42A	153	C_8H_{16} isomer
7	74	*i*-propanol	42B	153	$C_6H_{10}O$ isomer (tent.)
8A	76	methylene chloride	43A	155	C_8H_{14} isomer
8B	77	Freon 113	43B	156	$C_6H_{10}O$ isomer
9	79	carbon disulfide (trace)	44	158	unknown
10	80	C_4H_8O isomer	45A	159	ethylbenzene
11	85	$C_5H_{10}O$ isomer	45B	159	C_7H_{12} isomer (tent.)
12	86	*n*-butanal	45C	160	α-furfuryl alcohol
13A	57	methyl ethyl ketone	45D	161	xylene isomer
13B	88	C_6H_{14} isomer	45E	161	C_8H_{18} isomer
14A	90	hexafluorobenzene (int. std.)	46	164	$C_7H_{14}O$ isomer
14B	90	2-methylfuran	47	165	C_8H_{16} isomer
14C	90	*n*-hexane	48A	166	styrene
15A	92	unknown	48B	166	$C_7H_{10}O_2$ isomer
15B	93	3-methylfuran	48C	167	*n*-heptanal
16	94	C_6H_{12} isomer	49A	169	$C_6H_6O_2$ isomer
17A	97	perfluorotoluene (int. std.)	49B	170	unknown
17B	97	methylcyclopentane	50	172	*n*-nonane
18	98	C_4H_6O isomer	51A	173	C_8H_{16} isomer
19	100	1,1,1-trichloroethane	51B	173	C_8H_{14} isomer
20	104	benzene	52	175	unknown
21	106	C_6H_{12} isomer	53	176	C_9H_{18} isomer
22A	108	ethyl vinyl ketone	54	178	C_9H_{18} isomer
22B	108	$C_5H_{10}O$ isomer	55	180	$C_{10}H_{20}$ isomer
23	109	$C_6H_{12}O$ isomer	56	181	C_9H_{18} isomer (tent.)
24	110	*n*-pentanal	57A	182	methylfuraldehyde isomer
25A	113	C_7H_{14} isomer	57B	182	benzaldehyde
25B	113	trichloroethylene	58	184	methylfuraldehyde isomer
25C	114	C_6H_8O	59A	186	*n*-propylbenzene
26A	116	*n*-heptane	59B	187	$C_{10}H_{20}$ isomer
26B	117	acetic acid	59C	188	$C_{10}H_{22}$ isomer
27	120	2-vinylfuran	60	189	$C_{11}H_{24}$ isomer
28	122	C_7H_{14} isomer (tent.)	61	190	$C_9H_{18}O$ isomer
29	123	C_7H_{14} isomer	62A	191	unknown
30	125	dimethyl disulfide	62B	191	$C_{11}H_{22}$ isomer (tent.)
31	126	dihydropyran (tent.)	63A	192	$C_{11}H_{24}$ isomer
32	133	toluene	63B	193	2-pentylfuran
33	134	C_7H_{14} isomer	64	194	*n*-octanal

- continued -

Table D-7 (continued)

Chromatographic Peak No.	Elution Temp. (°C)	Compound
65A	194	C_3-alkyl benzene isomer
65B	194	unknown
66	196	siloxane
67A	197	*n*-decane
67B	198	dichlorobenzene
68	199	unsat. hydrocarbon
69	201	C_9H_{16} isomer
70	202	C_4-alkylbenzene (tent.)
71	204	$C_8H_6O_2$ isomer
72A	204	limonene
72B	204	sat. hydrocarbon
73A	207	unsat. hydrocarbon
73B	207	$C_{11}H_{24}$ isomer
74A	208	sat. hydrocarbon
74B	209	acetophenone
75	210	C_4-alkylbenzene
76	211	$C_{11}H_{24}$ isomer
77A	212	$C_{11}H_{24}$ isomer
77B	212	unsat. hydrocarbon
77C	213	sat. hydrocarbon
77D	213	$C_9H_8O_2$ isomer
78A	214	$C_7H_8O_2$ isomer (tent.)
78B	215	$C_{11}H_{24}$ isomer
79	217	$C_{10}H_{16}O$ isomer
80	218	*n*-nonanal
81	221	*n*-undecane
82	222	unsat. hydrocarbon
83	224	sat. hydrocarbon
84	226	$C_{12}H_{26}$ isomer
85	227	sat. hydrocarbon
86	228	$C_{12}H_{26}$ isomer
87A	229	siloxane
87B	231	$C_{12}H_{24}$ isomer
88A	234	siloxane
88B	234	$C_{10}H_{10}O_2$ (tent.)
89	235	sat. hydrocarbon
90	236	$C_{12}H_{26}$ isomer
91	237	$C_{12}H_{26}$ isomer
92A	238	$C_{10}H_{20}O$ isomer
92B	239	unsat. hydrocarbon
93A	240	naphthalene (trace)
93B	240	$C_{12}H_{22}$ isomer
94A	240	*n*-decanal
94B	240	$C_{12}H_{24}$ isomer
95	240	*n*-dodecane
96	240	$C_{13}H_{28}$ isomer
97	240	sat. hydrocarbon
98A	240	$C_{13}H_{26}$ isomer
98B	240	$C_{11}H_{20}O$ isomer
99	240	$C_{13}H_{28}$ isomer
100	240	$C_{13}H_{28}$ isomer
101	240	$C_{13}H_{28}$ isomer
102	240	$C_{10}H_{16}O$ isomer
103	240	$C_{13}H_{24}$ isomer
104	240	*n*-undecanal
105	240	*n*-tridecane
106	240	$C_{10}H_{16}O$ isomer
107	240	siloxane
108	240	unsat. hydrocarbon
109	240	unsat. hydrocarbon
110	240	*n*-dodecanal
111	240	*n*-tetradecane
112	240	unsat. hydrocarbon
113	240	*n*-pentadecane

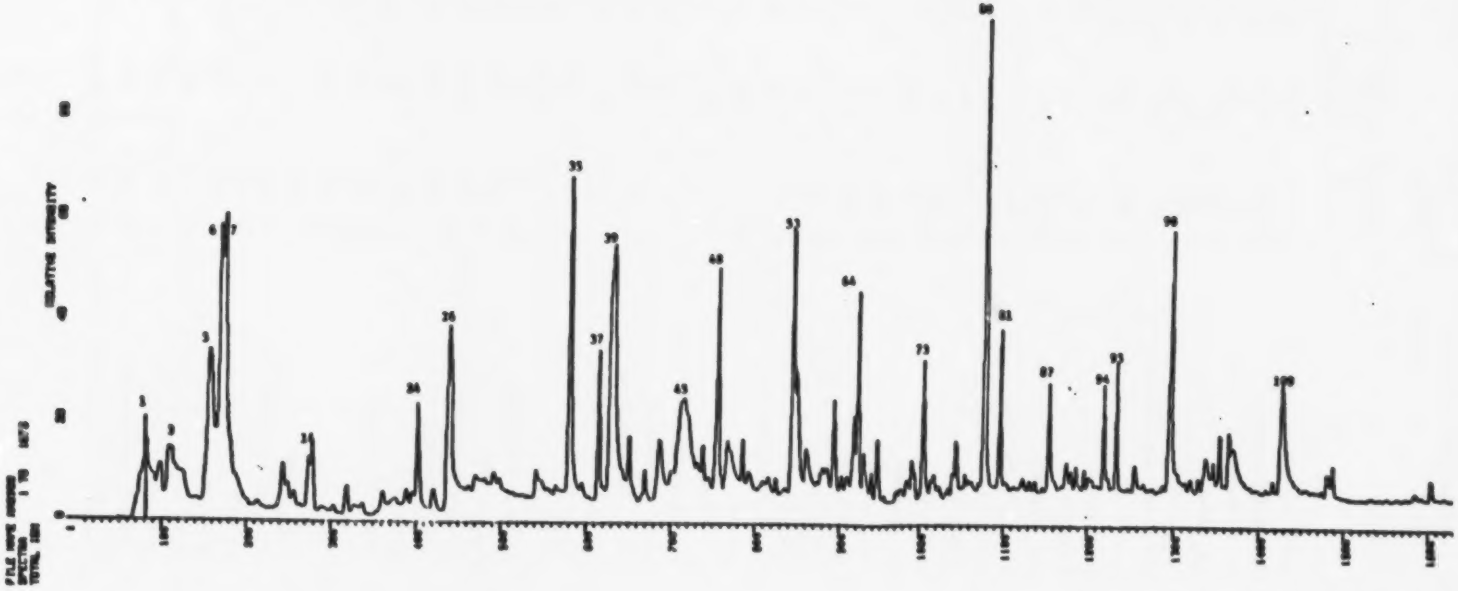

Figure D-7. Total ion current chromatogram from GC/MS analysis for volatiles in sample no. 3053 (Baton Rouge, LA).

Table D-8. VOLATILE COMPOUNDS IDENTIFIED IN PURGE OF SAMPLE NO. 3111 (Baton Rouge, LA)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
1	59	carbon dioxide
2	61	dichlorodifluoromethane
3A	65	sulfur dioxide
3B	65	C_4H_8 isomer
4	71	C_5H_{10} isomer
5A	73	trichlorofluoromethane
5B	74	acetone
6A	76	isopropanol
6B	76	*n*-pentane
6C	77	C_5H_8 isomer
7A	80	methylene chloride
7B	81	Freon 113
8	82	carbon disulfide
9	84	*n*-butanal
10A	87	cyclopentane
10B	88	C_6H_{14} isomer
11	89	$C_5H_{10}O$ isomer
12A	91	$C_5H_{10}O$ isomer
12B	92	C_6H_{12} isomer
13	94	hexafluorobenzene (int. std.)
14	95	*n*-hexane
15	96	chloroform
16A	101	perfluorotoluene (int. std.)
16B	101	methylcyclopentane
17A	104	1,1,1-trichloroethane
17B	104	$C_5H_{10}O$ isomer (tent.)
18	106	$C_6H_{12}O$ isomer.
19	108	benzene
20	109	carbon tetrachloride
21	110	C_6H_{12} isomer
22	111	$C_6H_{12}O$ isomer (tent.)
23	112	$C_6H_{12}O$ isomer (tent.)
24	114	*n*-pentanal
25	117	trichloroethylene
26	120	*n*-heptane
27	123	C_8H_{16} isomer
28	126	C_7H_{14} isomer
29	128	dimethyl disulfide
30	135	toluene
31A	142	*n*-hexanal
31B	144	C_8H_{16} isomer
32	146	*n*-octane
33A	148	tetrachloroethylene
33B	148	unsat. hydrocarbon
34	150	C_8H_{16} isomer (tent.)
35	152	siloxane
36A	155	C_9H_{18} isomer
36B	155	C_9H_{20} isomer (tent.)
37	161	ethylbenzene
38A	163	xylene isomer
38B	164	C_9H_{20} isomer
39A	168	styrene
39B	168	C_9H_{20} isomer
40	169	xylene isomer
41	170	C_9H_{20} isomer
42	173	C_9H_{20} isomer
43A	177	sat. hydrocarbon
43B	177	C_3-alkyl benzene (tent.)
44	178	$C_{10}H_{22}$ isomer
45	179	$C_{10}H_{22}$ isomer
46	181	sat. hydrocarbon
47	183	siloxane
48	186	benzaldehyde
49	189	unknown
50	189	$C_{11}H_{24}$ isomer
51	191	C_3-alkyl benzene
52	192	$C_{11}H_{24}$ isomer
53	193	$C_{11}H_{24}$ isomer
54A	194	$C_{11}H_{24}$ isomer
54B	195	C_3-alkyl benzene
55A	196	siloxane
55B	197	$C_{11}H_{24}$ isomer
56	198	dichlorobenzene
57	202	C_3-alkyl benzene
58	204	limonene
59	206	sat. hydrocarbon
60	208	sat. hydrocarbon
61A	212	acetophenone
61B	213	sat. hydrocarbon
62	214	sat. hydrocarbon
63	217	sat. hydrocarbon
64	221	*n*-undecane
65	233	siloxane
66	240	*n*-dodecane
67	240	unsat. hydrocarbon
68	240	siloxane

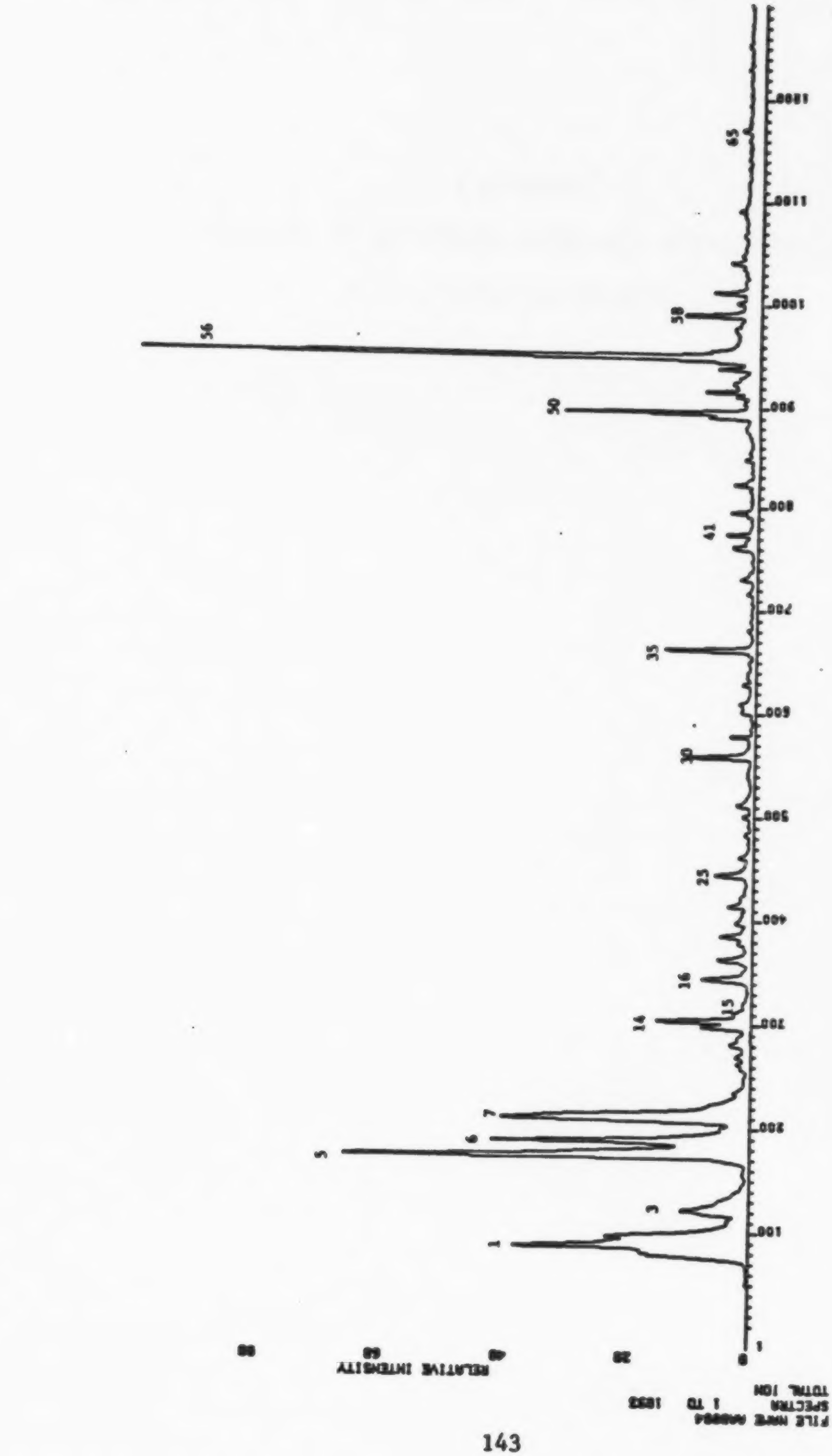

Figure D-8. Total ion current chromatogram from GC/MS analysis for volatiles in sample no. 3111 (Baton Rouge, LA).

APPENDIX E

SEMIVOLATILE COMPOUNDS IDENTIFIED IN SELECTED EXTRACTS OF MOTHER'S MILK

Table E-1. SEMIVOLATILE COMPOUNDS IDENTIFIED IN EXTRACT OF SAMPLE 1032 (Bayonne, NJ)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
1A		toluene
1B		xylene isomer
2		siloxane
3		siloxane
4		siloxane
5		siloxane
6		siloxane
7		siloxane
8		siloxane
9		dimethylbiphenyl (tent.)
10		siloxane
11A		siloxane
11B		unknown
12		siloxane
13		sat. hydrocarbon
14		siloxane
15		siloxane
16		sat. hydrocarbon
17		sat. and unsat. hydrocarbons
18		siloxane
19		siloxane
20		siloxane
21		sat. hydrocarbon
22		phthalate (tent.)
23		siloxane
24		sat. and unsat. hydrocarbons
25		unknown
26		unknown
27		siloxane
28		siloxane
29		d_{10}-pyrene (std.)
30		sat. and unsat. hydrocarbons
31		siloxane
32		DDE
33		unknown
34A		siloxane
34B		unknown
35		unknown
36		sat. and unsat. hydrocarbons
37		siloxane
38		sat. and unsat. hydrocarbons
39		sat. and unsat. hydrocarbons
40		siloxane
41		siloxane
42		siloxane
43		siloxane
44		siloxane
45		siloxane
46		lycopersene
47		cholesteryl acetate
48		siloxane

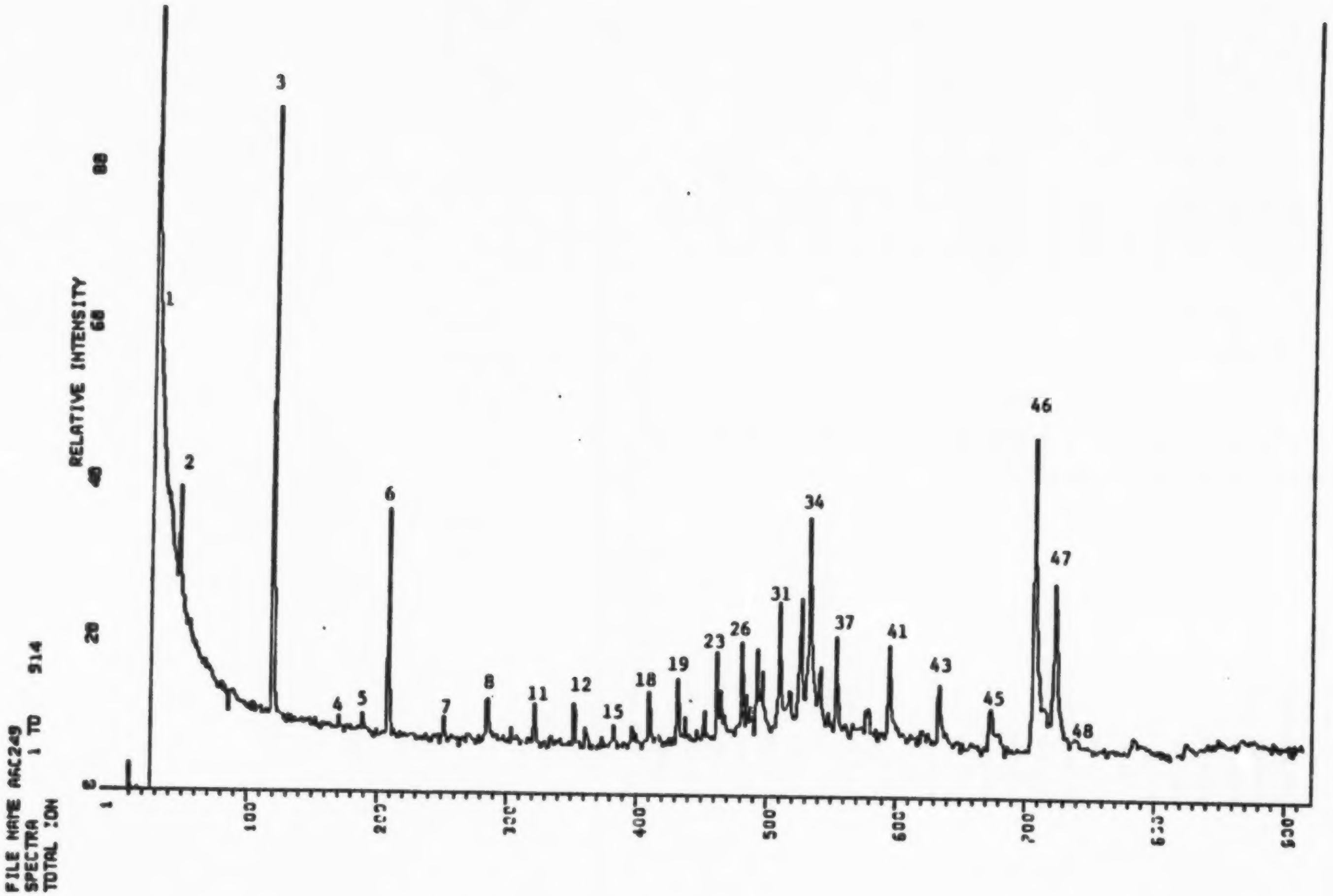

Figure E-1. Total ion current chromatogram from GC/MS analysis for Semivolatiles in sample 1032 (Bayonne, NJ).

Table E-2. SEMIVOLATILE COMPOUNDS IDENTIFIED IN EXTRACT OF SAMPLE 2121 (Pittsburgh, PA)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
1		toluene
2		siloxane
3		siloxane
4		siloxane
5		siloxane
6		2,6-di-tert-butyl-4-methylphenol
7		methyl dodecanoate
8		ethyl butyrate (tent.)
9		siloxane
10		sat. hydrocarbon
11		siloxane
12		siloxane
13		sat. hydrocarbon
14		siloxane
15		siloxane
16		siloxane
17		sat. and unsat. hydrocarbons
18		sat. hydrocarbon
19		unknown
20		siloxane
21		sat. and unsat. hydrocarbons
22		unknown
23		unknown
24		siloxane
25		siloxane
26		d_{10}-pyrene (int. std.)
27		siloxane
28		unsat. hydrocarbon
29A		unsat. hydrocarbon
29B		DDE
30		sat. and unsat. hydrocarbons
31		siloxane
32		pentachlorobiphenyl
33		sat. and unsat. hydrocarbons
34		siloxane
35		sat. and unsat. hydrocarbons
36		hexachlorobiphenyl
37		siloxane
38		sat. hydrocarbon
39		siloxane
40		sat. and unsat. hydrocarbons
41A		sat. and unsat. hydrocarbons
41B		heptachlorobiphenyl
42		siloxane
43		sat. and unsat. hydrocarbons
44		siloxane
45		siloxane
46		siloxane
47		siloxane
48		siloxane
49		lycopersene
50		siloxane
51		cholesteryl acetate

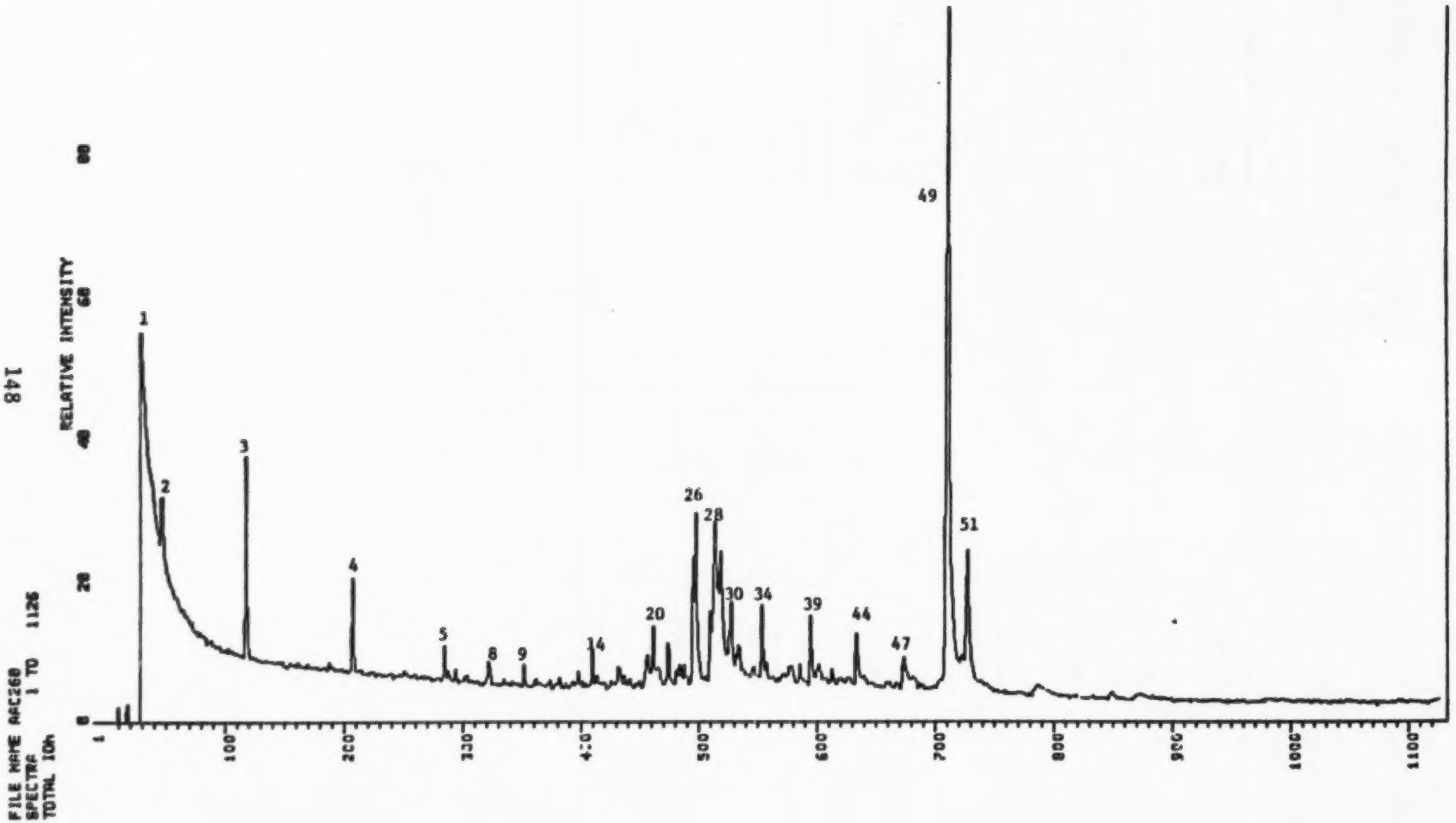

Figure E-2. Total ion current chromatogram from GC/MS analysis for semivolatiles in sample 2121 (Pittsburgh, PA).

Table E-3. SEMIVOLATILE COMPOUNDS IDENTIFIED IN EXTRACT OF SAMPLE 3095 (Baton Rouge, LA)

Chromatographic Peak No.	Elution Temp. (°C)	Compound
1		methylene chloride
2		toluene
3		siloxane
4		sat. hydrocarbon
5		sat. hydrocarbon (tent.)
6		siloxane
7		sat. hydrocarbon (tent.)
8		siloxane
9		sat. hydrocarbon (tent.)
10		siloxane
11		sat. hydrocarbon
12		sat. hydrocarbon
13		unknown
14		unknown
15		sat. hydrocarbon
16		siloxane
17		sat. hydrocarbon
18		siloxane
19		siloxane
20		sat. hydrocarbon
21		sat. hydrocarbon
22		siloxane
23		siloxane
24		siloxane
25		sat. hydrocarbon
26		siloxane
27A		sat. hydrocarbon
27B		unsat. hydrocarbon
28		unknown
29		unknown
30		siloxane
31		siloxane
32		d_{10}-pyrene
33A		sat. hydrocarbon
33B		unsat. hydrocarbon
34		siloxane
35		DDE
36A		unknown
36B		unsat. hydrocarbon
37A		siloxane
37B		unknown
38		sat. hydrocarbon (tent.)
39		siloxane
40		unsat. hydrocarbon (tent.)
41		siloxane
42		sat. hydrocarbon (tent.)
43		siloxane
44		sat. hydrocarbon
45		sat. hydrocarbon
46A		sat. hydrocarbon
46B		siloxane
47		siloxane
48		sat. hydrocarbon
49		siloxane (tent.)
50A		siloxane
50B		sat. hydrocarbon
51		sat. hydrocarbon
52		lycopersene
53A		siloxane
53B		cholesteryl acetate
54		siloxane
55		sat. hydrocarbon
56		unknown
57		siloxane

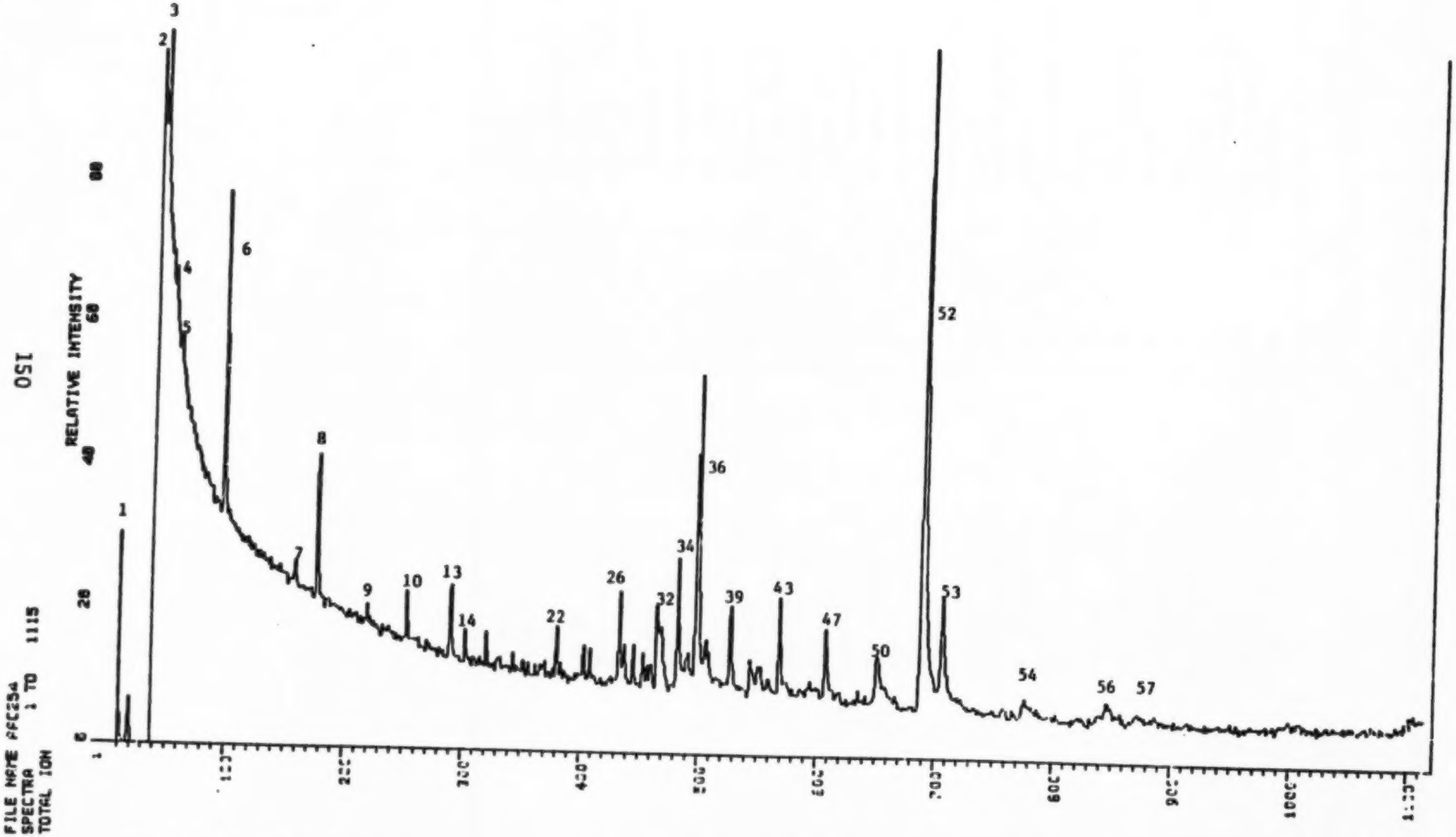

Figure E-3. Total ion current chromatogram from GC/MS analysis for semivolatiles in sample 3095 (Baton Rouge, LA).

Table E-4. SEMIVOLATILE COMPOUNDS IDENTIFIED IN EXTRACT OF SAMPLE 4093 (Charleston, WV)

Chromato-graphic Peak No.	Elution Temp. (°C)	Compound	Chromato-graphic Peak No.	Elution Temp. (°C)	Compound
1		toluene	30		siloxane
2		siloxane	31		siloxane
3		siloxane	32		d_{10}-pyrene (int. std.)
4A		siloxane	33		sat. and unsat. hydrocarbons
4B		sat. hydrocarbon	34		siloxane
5		siloxane	35		sat. and unsat. hydrocarbons
6		siloxane	36		sat. and unsat. hydrocarbons
7		butyric anhydride (tent.)	37A		sat. and unsat. hydrocarbons
8		sat. hydrocarbon	37B		DDE
9A		C_9H_{20} isomer	38		sat. and unsat. hydrocarbons
9B		unknown	39		siloxane
10		siloxane	40		siloxane
11		sat. hydrocarbon	41		sat. and unsat. hydrocarbons
12		sat. hydrocarbon	42A		siloxane
13		siloxane	42B		methyl dehydroabietate (tent.)
14		siloxane	43		siloxane
15		sat. hydrocarbon	44		sat. hydrocarbon
16		sat. hydrocarbon	45		siloxane
17		sat. hydrocarbon	46		sat. and unsat. hydrocarbons
18		sat. hydrocarbon	47		siloxane
19		unknown	48		phthalate
20		siloxane	49		siloxane
21		sat. hydrocarbon	50		unknown
22		sat. and unsat. hydrocarbon	51		siloxane
23A		siloxane	52		siloxane
23B		sat. and unsat. hydrocarbons	53		siloxane
24		siloxane	54		lycopersene
25		sat. and unsat. hydrocarbons	55		siloxane
26		siloxane	56		cholesteryl acetate
27		unknown	57		sat. and unsat. hydrocarbons
28		sat. and unsat. hydrocarbons	58		siloxane
29		sat. and unsat. hydrocarbons	59		α-tacopherol (vitamin)

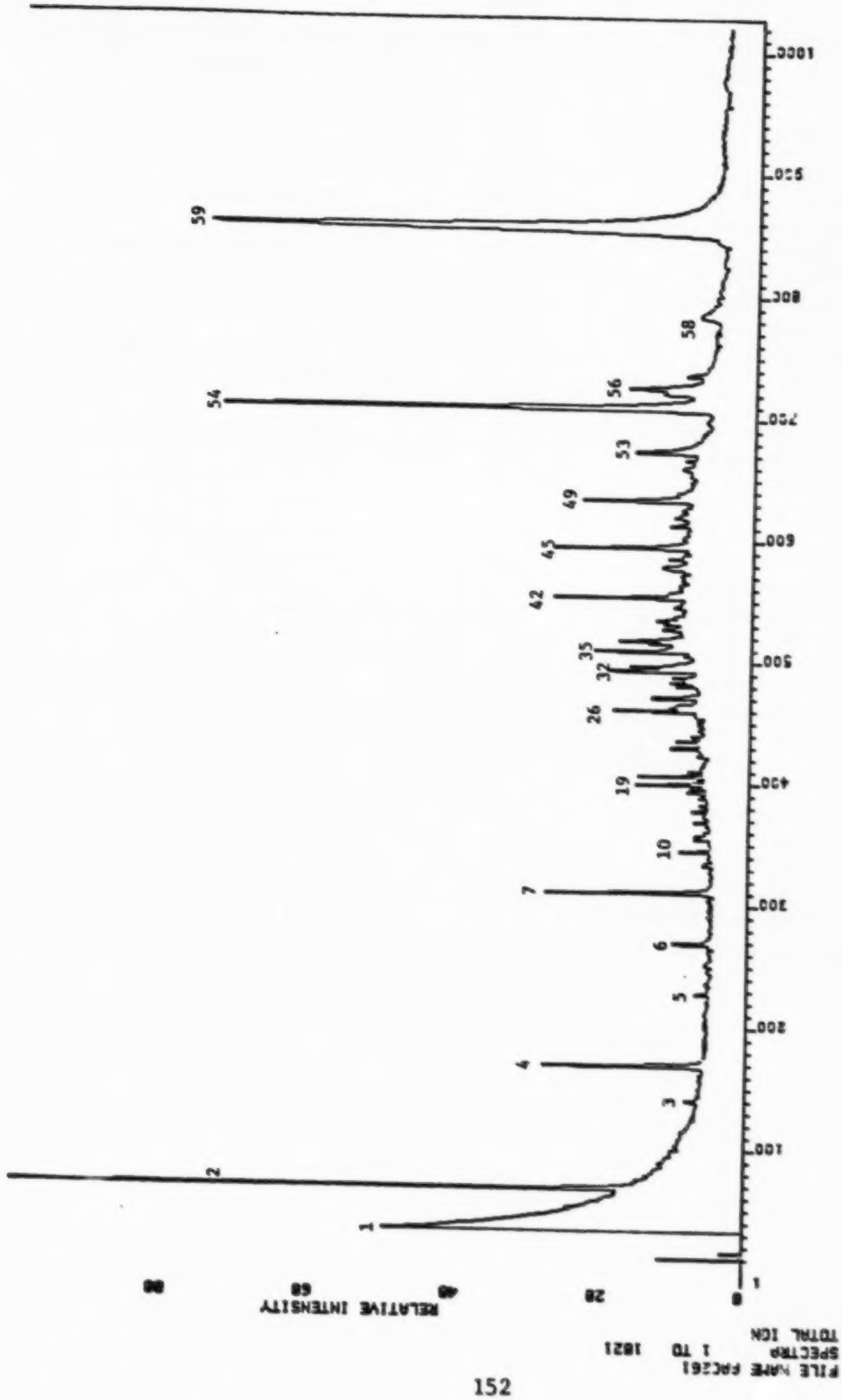

Figure E-4. Total ion current chromatogram from GC/MS analysis for semivolatiles in sample 4093 (Charleston, WV).